

AD-A188 361

DTIC FILE COPY (12)

ANALYSIS OF MODEL FUELS AND THEIR COMPATIBILITY WITH SELECTED ELASTOMERS – PART I

INTERIM REPORT
BFLRF No. 238

By

G.E. Fodor
B.K. Bailey
D.L. Present
K.B. Kohl

Belvoir Fuels and Lubricants Research Facility (SwRI)
Southwest Research Institute
San Antonio, Texas

Under Contract to

U.S. Army Belvoir Research, Development
and Engineering Center
Materials, Fuels and Lubricants Laboratory
Fort Belvoir, Virginia

Contract No. DAAK70-85-C-0007

Approved for public release; distribution unlimited

September 1987

DTIC
ELECTE
NOV 20 1987
S D

87 11 9 012

Disclaimers

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Trade names cited in this report do not constitute an official endorsement or approval of the use of such commercial hardware or software.

DTIC Availability Notice

Qualified requestors may obtain copies of this report from the Defense Technical Information Center, Cameron Station, Alexandria, Virginia 22314.

Disposition Instructions

Destroy this report when no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS None	
2a. SECURITY CLASSIFICATION AUTHORITY N/A			3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A				
4. PERFORMING ORGANIZATION REPORT NUMBER(S) Interim Report BFLRF No. 238			5. MONITORING ORGANIZATION REPORT NUMBER(S)	
6a. NAME OF PERFORMING ORGANIZATION Belvoir Fuels and Lubricants Research Facility (SwRI)		6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION	
6c. ADDRESS (City, State, and ZIP Code) Southwest Research Institute 6220 Culebra Road San Antonio, Texas 78284			7b. ADDRESS (City, State, and ZIP Code)	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Belvoir Research, Development and Engineering Center		8b. OFFICE SYMBOL (If applicable) STRBE-VF	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER DAAK70-85-C-0007; WD 16	
8c. ADDRESS (City, State, and ZIP Code) Fort Belvoir, VA 22060-5606			10. SOURCE OF FUNDING NUMBERS	
			PROGRAM ELEMENT NO. 63104D	PROJECT NO. 1L2631- 04D150
			TASK NO. 08	WORK UNIT ACCESSION NO.
11. TITLE (Include Security Classification) Analysis of Model Fuels and Their Compatibility With Selected Elastomers--Part I (U)				
12. PERSONAL AUTHOR(S) Fodor, George E., Bailey, Brent K.; Present, David L.; and Kohl, K.B.				
13a. TYPE OF REPORT Interim		13b. TIME COVERED FROM Mar 87 TO Sep 87		14. DATE OF REPORT (Year, Month, Day) 1987 September
15. PAGE COUNT 64				
16. SUPPLEMENTARY NOTATION				
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD GROUP SUB-GROUP			Diesel fuel, Chemical analysis, GC/MS Elastomer, Thermal Oxidation test, ← Compatibility, NMR	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) In this program, three diesel fuels and a diesel fuel-blending component have been analyzed to initiate development of a diesel fuel compositional data base to be used to (a) design a repeatable quality, model compound based diesel fuel simulant for the evaluation of fuel-elastomer compatibility, and (b) aid in understanding and ultimately controlling fuel-elastomer interactions. The initial results of compatibility studies between fresh and thermally aged diesel fuels, and four elastomer-based fuel tank materials are summarized in this report. (Key words)				
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED//UNLIMITED <input type="checkbox"/> SAME AS RPT. <input type="checkbox"/> DTIC USERS			21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL Mr. F.W. Schaeckel			22b. TELEPHONE (Include Area Code) (703) 664-3576	
			22c. OFFICE SYMBOL STRBE-VF	

FOREWORD

This work was conducted at the Belvoir Fuels and Lubricants Research Facility (SwRI), Southwest Research Institute, under DOD Contract No. DAAK70-85-C-0007. The project was administered by the Fuels and Lubricants Division, Materials, Fuels and Lubricants Laboratory, U.S. Army Belvoir Research, Development and Engineering Center, Fort Belvoir, Virginia 22060-5606, with Mr. F.W. Schaeckel, STRBE-VF, serving as Contracting Officer's Representative. This report covers the period of performance from March 1987 to September 1987.



Accession For	
NTIS CRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By	
Distribution/	
Availability Codes	
Dist	Avail And/or Special
A-1	

ACKNOWLEDGEMENTS

Project planning guidance was provided by Messrs. M.E. LePera, P. Touchet, F.W. Schaekel, and Dr. J. Mengenhauser of the U.S. Army Belvoir Research, Development and Engineering Center, and by Mr. S.J. Lestz of the Belvoir Fuels and Lubricants Research Facility (SwRI).

The invaluable contributions by Dr. N.F. Swynnerton of Southwest Research Institute to the nuclear magnetic resonance analyses of the fuels are gratefully acknowledged. The JFTOT work was supervised and interpreted by J.A. Russell. Laboratory assistance was provided by M. Voigt, R. Robledo, T. Saucedo, V. Barnhill, J. Dozier, R. Powell, and G. Segura. Editorial assistance by J. Pryor, C. Sturrock, S. Douvry, and L. Pierce is also acknowledged with appreciation.

TABLE OF CONTENTS

<u>Section</u>	<u>Page</u>
I. INTRODUCTION	1
II. OBJECTIVE	1
III. APPROACH	1
IV. SELECTION AND PROCUREMENT OF FUELS	2
V. ANALYSIS OF FUELS	3
A. Specification-Type Analyses	3
B. Nuclear Magnetic Resonance Analysis	6
C. GC/MS Analysis of Model Fuels	15
VI. FUEL-ELASTOMER COMPATIBILITY STUDIES	18
VII. CONCLUSIONS AND RECOMMENDATIONS	24
VIII. LIST OF REFERENCES	25
APPENDIX	
Detailed GC/MS Data	27

LIST OF ILLUSTRATIONS

<u>Figure</u>	<u>Page</u>
1 Examples for NMR Assignments	8
2 Spun TDR Ratings for all Fuel-Elastomer-Aging Combinations	22

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1 Analysis of Model Fuels	4
2 Proton Chemical Shift Correlation Chart for Hydrocarbons According to Gillet	7
3 ¹³ C Chemical Shift Correlation Chart for Hydrocarbons According to Gillet	7
4 Proton and ¹³ C NMR Data on Middle Distillates (Gillet's Method)	9
5 Analysis of Middle Distillates by Nuclear Magnetic Resonance Spectroscopy (Gillet's Method)	10
6 NMR Chemical Shift Correlations (Bailey's Method)	12
7 Average Molecular Parameter Calculations (Bailey's Method)	12
8 Nuclear Magnetic Resonance Analysis (Bailey's Method)	13
9 Calculated Average Molecular Parameters (Bailey's Method)	14
10 Gas Chromatographic Conditions for the Analysis of Fuel Fractions	16
11 Mass Spectrometer Operating Conditions	16
12 GC/MS Analysis of the Middle Distillate Products	17
13 Determination of Conditions for Preoxidation	19
14 Evaluation of the Effects of Elastomers on Unaged and Aged Fuels	21

I. INTRODUCTION

According to U.S. Army field experience, diesel fuels attack collapsible fuel tanks, especially when the diesel fuel is spilled on the surface of these tanks.(1-7)* Shertzer (8) and Hazlett (9) have shown that peroxides in turbine fuels attack certain elastomers used in jet engine fuel pumps. Russell (10,11) stated that high aromatic hydrocarbon concentrations in fuels may cause severe swelling of certain elastomers. Existing specifications, e.g., federal specification TT-S-735 and its proposed modification TT-S-735A, do not have recommendations for fluids to simulate the effects of middle distillate fuels, such as the various grades of diesel fuels and the lower volatility turbine fuels. In fact, TT-S-735A does not address the boiling range between JP-4 and light mineral oil. This project addresses this deficiency.

II. OBJECTIVE

The objective of this project is to provide a basis for improving the storage life of collapsible tanks and other fuel-handling equipment exposed to DF-2 type middle distillate fuels. This objective is to be achieved through the selection and analysis of model fuels that may yield a consistent, reproducible middle distillate fuel simulant for the evaluation of fuel-elastomer interactions.

III. APPROACH

Project goals were to be addressed in three phases. The goal of Phase I was to begin to develop a synthetic test fluid blend of reproducible composition to simulate the effects of middle distillate fuels on elastomeric, collapsible fuel tanks. Phase II was to evaluate the effects of specific elastomers on selected distillate fuels, while studies under Phase III were to address the improvement of test methodology for determining compatibility of fuels with elastomers. An additional goal of Phase III was to isolate and study the extracts from several elastomers using a variety of solvents and the synthetic middle distillate fluid simulant developed under Phase I. However, Phase III is being delayed until Phases I and II provide sufficient information.

* Underscored numbers in parentheses refer to the list of references at the end of this report.

During initiation of the project, fuel samples were procured and analyzed. Additionally, compatibility studies of unaged and aged (i.e., partially oxidized) fuels with selected elastomers were begun. This report addresses partial objectives of Phases I and II, which were performed during the first 6-month time frame.

IV. SELECTION AND PROCUREMENT OF FUELS

Three diesel fuels and a diesel fuel blending stock were procured. The selection was based on experience and technical judgement. These products included representative samples of the following items:

- (a) A Caterpillar 1H2/1G2 reference fuel (No. 16058) is used in determining the effects of lubricating oils on ring sticking, ring and cylinder wear, and accumulation of piston deposits. The use of this fuel is required in the following standard lubricant tests, monitored and approved by ASTM Test Monitoring Center:
 - (1) Caterpillar 1G2, 1H2, OL-5, OL-6
 - (2) Cummins NTC-400
 - (3) U.S. Army 6V53T (FTM 354 and 355)
- (b) A Phillips 2-D diesel control fuel (No. 15978), the required EPA endurance test fuel used in emission certification testing of light-duty and heavy-duty diesel engines as specified in CFR 86.113-82(b)(2), and CFR 86.1313-84(b)(2), respectively. The specifications require a minimum aromatic hydrocarbon content of 27 vol% as measured by ASTM D 1319.
- (c) A modified MIL-F-46162C(ME) referee grade diesel fuel (No. 16082). This marginal quality, 1-percent sulfur-containing fuel was designed to be used in research, development, and proof testing of compression ignition engines and other equipment that are to operate with tactical grades of diesel fuels conforming to VV-F-800 specifications. As it is expected that products of fuel oxidation may be prime contributors to elastomer deterioration, exclusion of the stabilizer additive was specified to allow unimpeded, but controlled fuel oxidation. Further, since a requirement for fuel system icing inhibitor (FSII) became part of the procurement action for the development

of new collapsible tank materials, it was specified that this fuel should contain 1 vol% of ethylene glycol monomethyl ether as FSII;

- (d) The selected diesel fuel blending stock was a "typical" light cycle oil (LCO) (No. 15980), having characteristically high susceptibility to oxidation, and high concentrations of aromatic hydrocarbons.

To stabilize the procured fuels in their epoxy-lined drums upon receipt, each fuel was "inerted" by purging with argon. For immediate use, a 5-gallon sample of each fuel was withdrawn into an epoxy-lined can. These fuel samples have been kept under an argon atmosphere in cold storage at about 5°C.

V. ANALYSIS OF FUELS

Detailed analyses were performed on each of the four middle distillate products (a) to help define a fuel simulant composition that would best serve the study of such fuel's compatibility with collapsible elastomeric fuel tank material, and (b) to aid in understanding fuel-elastomer interactions.

A. Specification-Type Analyses

Analysis of the fuels included specification tests (ASTM) and special methodology. The specification type data are given in TABLE 1. Special attention is directed to several aspects of the analytical data presented in TABLE 1.

It may be noted that, although the variations in the boiling ranges of these products are relatively large, the average molecular weights, as measured by the various methods, are fairly close, ranging between 197 and 224 grams per mole.

It was not surprising that the LCO (Fuel No. 15980) showed appreciably higher carbon-to-hydrogen ratio than the three diesel fuels. For the three diesel fuels, the average value of C/H is 6.83, while it is 8.34 for the light cycle oil. This increased C/H ratio is due to the increased aromatic hydrocarbon content of this product. Further evidence for this fact may be found in aromatic ring carbon content measurements by ultraviolet spectrophotometry, high-performance liquid chromatography (HPLC) data, nuclear magnetic resonance (NMR) spectra, and GC/MS results.

TABLE 1. Analysis of Model Fuels

Property	Method	Fuel Identification Number			
		16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
Gravity, °API	D 1298	34.6	35.4	29.1	20.8
Density, kg/L at 15°C	D 1298	0.8515	0.8478	0.8806	0.9286
Color	D 1500	1.0	L2.5	L1.0	2.0
Flash Point, PMCC, °C	D 93	86	64	57	112
Cloud Point, °C	D 2500	-11	-14	+15	-2
Cloud Point, °C, CaCl ₂ dried fuel		-11	-15	-15	-2
Pour Point, °C	D 97	-12	-17	-15	-3
Pour Point, °C, CaCl ₂ dried fuel		-14	-19	-18	-8
Kinematic Viscosity, cSt,					
at 20°C	D 445	5.04	3.70	4.93	7.40
at 40°C	D 445	3.13	2.40	2.98	4.14
Distillation, °C	D 86				
IBP		210	187	149	240
10% Recovered		243	218	223	262
50% Recovered		274	260	280	297
90% Recovered		314	306	330	328
EP		343	337	355	362
Recovered, Vol%		99	99	99	99
Residue, Vol%		1	1	1	1
Ash, wt%	D 482	<0.01	<0.01	<0.01	<0.01
Carbon Residue, 10% Bottoms, wt%	D 524	0.12	0.12	0.11	0.23
Particulate Contamination, mg/L	D 2276	0.9	2.0	3.0	0.3
Accelerated Stability, mg/100 mL	D 2274	1.1	0.1	0.3	0.3
Existent Gum, mg/100 mL	D 381	4.5	5.9	3.0	7.1
Thermal Stability, JFTOT, at 260°C	D 3241				
Visual Rating		2	1	3	1
ΔP, mm Hg		0	0	0	0
Neutralization No., mg KOH/g	D 664	0.04	0.01	0.04	0.01
Copper Strip Corrosion	D 130	1A	1A	1A	1A
Carbon, wt%		86.41	86.61	86.11	88.75
Hydrogen, wt%		12.97	12.89	12.08	10.64
Nitrogen, ppm	D 4629	69	580	8	760
Sulfur, wt%		0.42	0.36	1.02	0.50
Bromine Number, g/100 g	D 1159	1.8	1.9	3.6	10.1
Molecular Weight, Average					
VPO		209	224	221	216
UOP		212	197	201	209
API		218	202	209	216
Water, ppm	D 1744	208	70	257	344
Polars, wt%	HPLC	25.7	33.0	44.5	65.3
Nonpolars, wt%	HPLC	74.3	67.0	55.5	34.7
Aromatics, Ring Carbon, wt%	UV				
Mononuclear		6.0	7.2	14.4	7.3
Dinuclear		7.4	7.1	9.1	18.1
Trinuclear		1.1	0.8	0.8	5.6
Total		14.5	15.1	24.3	31.0
Peroxide No., ppm	D 3703	1.6	0.0	1.3	2.0
Heat of Combustion, net	D 240				
Btu/lb		18,260	18,160	17,828	17,624
MJ/kg		42.47	42.24	41.47	40.99
Cetane Number	D 613	48.6	45.3	42.7	-
Cetane Index		48.2	46.4	40.8	32.3

Thermal stability data, as determined by the JFTOT (ASTM D 3241) procedure at 260°C, are presented in TABLE 1. For subsequent studies, the breakpoint temperature for each of these products was determined. The JFTOT breakpoint is defined as the "incipient visual code 3 temperature," that is determined within a range of $\pm 5^\circ\text{C}$. The breakpoint temperatures were found to be 264°C, 279°C, 267°C, and 277°C, for fuel numbers 16058 (Cat), 15978 (EPA), 16082 (Ref), and 15980 (LCO), respectively. Based upon these findings, further JFTOT experiments on these fuels were carried out at 279°C.

Bromine number of an organic compound is normally a measure of its unsaturation. (Some phenols and nitrogen compounds may cause interference.) From such data, the olefin concentration of a fuel may be calculated by assuming that the average molecular weight of the olefins in the fuel is the same as that of the fuel, and that there is only one carbon-carbon double bond present per molecule. With these assumptions, the olefin content of the four products were estimated to be 2.4, 2.7, 5.0, and 13.7 wt% for fuel numbers 16058 (Cat), 15978 (EPA), 16082 (Ref), and 15980 (LCO), respectively.

The average molecular weight of each of the products, using three different techniques, is also presented in TABLE 1. Vapor pressure osmometry (VPO) is a direct laboratory measurement; however, it is restricted to fuels having an initial boiling point above 220°C. The only fuel in this category is Fuel No. 15980, the LCO. The other methods to determine average molecular weights of fuels, those by Universal Oil Products (UOP Method 375-59) and the American Petroleum Institute (API Technical Data Book 2B6.1), are calculation techniques, using fuel density and boiling point distribution. Both the UOP and API methods are used extensively in the petroleum industry for fuel characterization and process design work. Further calculations were made by taking the numeric average of the UOP and API molecular weight data.

While identification of individual compounds in complex mixtures, e.g., petroleum products, may be impossible, it was attempted to give as near a full analysis to these products as state-of-the-art technology permitted. To obtain chemical compositional details on the three diesel fuels and on the LCO, proton and ^{13}C nuclear magnetic resonance (NMR) spectra were obtained, and high-resolution gas chromatographic analyses were performed using mass spectroscopic detection (GC/MS). In the following sections, the results of these studies are summarized.

B. Nuclear Magnetic Resonance Analysis

Nuclear magnetic resonance spectroscopy was performed on a JEOL Model FX-90Q Fourier transform broad-band multinuclear instrument, operated according to the procedures of Gillet, Rubini, Delpuech, et al.(12) Deuteriochloroform, CDCl_3 , was used as the solvent, p-dioxane was the internal standard, and ferric acetylacetonate ($\text{Fe}(\text{acac})_3$) served as the relaxation agent in the ^{13}C experiments. In addition to developing interpretation of the NMR spectra according to the method of Gillet, et al., an alternative interpretation of the same spectra, according to the recommendations of Bailey and co-workers (13,14) is also offered. This alternative method uses slightly modified chemical shift assignments and fewer assumptions.

1. NMR Interpretations According to Gillet

Correlation charts for proton and ^{13}C from Reference 8 are reproduced in TABLES 2 and 3, respectively. Examples of the various NMR assignments are given in Fig. 1. The measured NMR integration data, grouped according to the recommendations of Gillet, are given in TABLE 4. From the data presented in TABLE 4, calculations were performed, and interpretations were derived by the method of Gillet to yield the results summarized in TABLE 5.

While the actual numerical data derived by the various analytical methods are not expected to yield the same data, directionally the NMR data may be used to complement the ASTM type, HPLC and ultraviolet spectroscopic data, with the NMR data providing more chemical structural details.

From the analyses presented thus far, it may be concluded that the order of increasing aromaticity is Fuel No. 16058 (Cat 1H2/1G2), No. 15978 (EPA-2D), No. 16082 (referee grade), and finally Fuel No. 15980, the light cycle oil. The fact that the NMR data-derived "aromaticity factors" are nearly identical to the values for aromatic and polar content as determined by HPLC, may be partly due to coincidence. It was shown by Fodor (15) that any compound having at least one aromatic ring with alkyl substitution not higher than C-12 will register as an aromatic hydrocarbon by this HPLC analysis, while ^{13}C NMR actually "counts" the carbon atoms in the various chemical structures. The relationship between the ^{13}C NMR data and the UV-derived aromatic ring carbon

**TABLE 2. Proton Chemical Shift Correlation Chart
for Hydrocarbons According to Gillet**

Shift Range (ppm from TMS)	Assignment	
9.30-6.30	H _{ar}	aromatic protons
6.30-4.50	H _o	olefinic protons
4.50-1.85	H _α (sat)	protons attached to a saturated α-carbon
1.85-1.00	H _β (sat)	protons attached to a saturated β-carbon (CH ₂) or to a β,γ,δ or more) carbon* (CH ₂ ,CH)
1.00-0.50	H _γ (sat)	methylic protons on a γ,δ or more) carbon*

*With respect to an eventual aromatic ring

**TABLE 3. ¹³C Chemical Shift Correlation Chart
for Hydrocarbons According to Gillet**

Shift Range (ppm from TMS)	Assignment	
0-70.0	C _{al}	aliphatic carbons
20.0-45.0	C _n	naphthenic carbons
118.0-130.5	C _{ar,H}	aromatic protonated carbons
123.5-126.0	C _{ar,ar,ar}	carbons at the junction of three aromatic rings
128.5-136.0	C _{ar,ar}	carbons at the junction of two aromatic rings
129.0-137.0	C _{ar,CH₃}	methyl-substituted aromatic carbons
132.0-137.0	C _{ar,n}	carbons at the junction of an aromatic and a naphthenic ring
137.0-160.0	C _{ar,alk}	alkyl-substituted (methyl group excluded) aromatic carbons

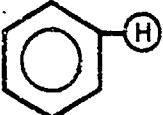
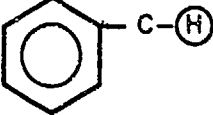
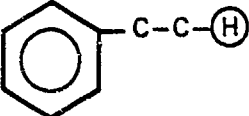
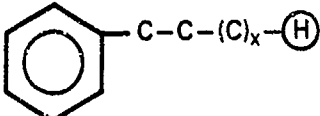
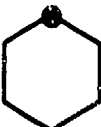

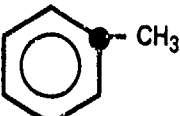

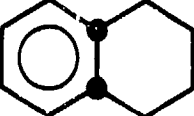

DESCRIPTION OF H OR C	ABBREVIATIONS	EXAMPLES
AROMATIC PROTONS	H (AR)	
PROTONS ON ALPHA CARBONS	H (α)	
PROTONS ON BETA CARBONS	H (β)	
PROTONS ON "OUTER" CARBONS	H (γ)	
ALIPHATIC CARBONS	C (SAT)	$R-\text{C}-R'$
NAPHTHENIC CARBONS	C (N)	
AROMATIC PROTONATED CARBONS	C (AR,H)	
METHYL SUBSTITUTED AROMATIC CARBONS	C (AR,CH ₃)	
ALKYL SUBSTITUTED AROMATIC CARBONS	C (AR,ALK)	
CARBONS AT AROMATIC-NAPHTHENIC JUNCTIONS	C (AR,N)	
CARBONS AT AROMATIC-AROMATIC JUNCTIONS	C (AR,AR)	

Figure 1. Examples for NMR Assignments

**TABLE 4. Proton and ^{13}C NMR Data on Middle Distillates
(Gillet's Method)**

^1H Integration Shift Range (ppm from TMS)	Fuel Identification			
	16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
9.30-6.30 aromatic protons	8.0	11.0	11.0	24.0
6.30-4.50 olefinic protons	0.0	0.0	0.0	0.0
4.50-1.85 H on sat. alpha C	9.0	14.0	24.0	33.0
1.85-1.00 H (beta)	96.0	89.5	87.0	68.0
1.00-0.50 H (gamma)	49.0	52.0	44.0	29.0
Reference (p-dioxane)	21.0	17.0	24.0	23.0
Integration sum, less reference	162.0	166.5	166.0	154.0
^{13}C Integration Shift Range (ppm from TMS)				
118.0-160.0 C (ar)	23.5	28.0	41.0	57.0
137.0-160.0 C (ar,alk)	4.0	5.5	6.5	6.7
132.0-137.0 C (ar,n)	3.5	4.5	8.0	8.5
129.0-137.0 C (ar,methyl)	6.5	7.5	11.0	15.5
128.5-136.0 C (ar,ar)	6.5	5.5	11.5	15.5
123.5-126.0 C (ar,ar,ar)	5.0	5.5	8.5	13.0
118.0-130.5 C (ar,H)	14.0	17.0	25.0	35.5
118.0-137.0 C (ar) less C (ar,alk)	19.5	23.0	35.0	49.0
0.0-70.0 C (al)	119.0	113.5	128.5	85.5
(14.5-16.9) + (18.5-21.0 Methyl (ar)	11.0	10.0	13.5	10.0
20.0-45.0 C (n)	95.0	87.5	98.0	63.5
Reference (p-dioxane)	15.0	15.5	18.7	14.0
Integration sum, less reference	142.5	141.5	169.5	142.5

TABLE 5. Analysis of Middle Distillates by Nuclear Magnetic Resonance Spectroscopy (Gillet's Method)

Assignment of Protons, wt%	Fuel Identification			
	16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
H (aromatic)	4.9	6.6	6.6	15.6
H (olefinic)	0.0	0.0	0.0	0.0
H (saturated)	95.1	93.4	93.4	84.4
H (alpha, saturated)	5.6	8.4	14.5	21.4
H (beta, saturated)	59.3	53.8	52.4	44.2
H (gamma, saturated)	30.2	31.2	26.5	18.8
Assignment of Carbons, wt%				
C (aromatic)	16.5	19.8	24.2	40.0
C (ar,alk, methyl excluded)	2.8	3.9	3.8	4.7
C (ar, methyl substituted)	4.6	5.3	6.5	10.9
C (ar, H)	9.8	12.0	14.7	24.9
C (aliphatic)	16.8	18.4	18.0	15.4
C (naphthenic)	66.7	61.8	57.8	44.6
C (saturated)	83.5	80.2	75.8	60.0
Methyl, gamma	49.1	67.0	40.5	24.8
Structural Parameters				
C/H (ratio of atoms)	0.616	0.466	0.655	0.760
C/H (weight ratio)	7.39	5.59	7.86	9.12
Aromaticity Factor	0.28	0.32	0.42	0.66
Mean Length of Aliphatic Chain	30	21	20	13
Branching Index	18	17	11	5

content should give similar data, as they both measure carbon atoms within aromatic ring systems. Data derived by the two systems give directionally similar results for the four fuels.

Comparison of the carbon-to-hydrogen weight ratios, as measured by elemental analysis and by nuclear magnetic resonance spectroscopy, shows similar results. Both methods give highest C/H ratio for the LCO, the most aromatic of the four "model" products. The NMR data-derived "mean length of aliphatic chain" is probably in error, as it is impossible to have an alkyl chain containing over 20 carbon atoms, while having an average molecular weight in the range of 197 to 224.

The fact that NMR did not detect olefins in any of the products, while HPLC detected trace concentrations of olefins in the LCO, and the bromine number determination gave positive values for the presence of carbon-carbon double bonds in each of the fuels indicate the uncertainty associated with the various methodologies in attempting to determine low concentrations of given species. It should also be mentioned that integration of an NMR spectrum is normally considered to have an error of about 5 percent. Signal overlap and assumptions may lead to substantially greater errors.

As Gillet, et al. (12) caution, NMR assignments for naphthenic compound types may only be considered "very approximate estimations" due to the broad envelope of the C-13 spectrum produced by cyclo-paraffins, upon which are superimposed among the relatively sharp lines of the normal paraffins. The other assignments of the NMR interpretations use only a minimum number of preliminary hypotheses, keeping reliability of the estimates normally to within 5 percent.

2. NMR Interpretations According to Bailey

An alternative NMR analysis of the same spectra of the four test fuels may be made according to the method of Bailey and Dalling.(13,14) In comparison with the previously discussed interpretation, this method uses somewhat modified chemical shift assignments and less assumptions. Their chemical shift assignments and average molecular parameter calculations are summarized in TABLES 6 and 7.

For these slightly modified assignments, the proton NMR integrals were remeasured. The results of this analysis are presented in TABLE 8. All proton and ^{13}C results are in agreement in ranking the aromaticity of the four products, supporting the results of the HPLC and the UV-derived aromatic ring carbon measurements.

Several regions of the proton NMR spectra and their respective percentages are presented in TABLE 8. As indicated by the descriptions, there is an overlap in three out of five saturate proton assignment areas. The two without overlap are the alpha methyl and the gamma methyl regions. These regions may be reliably used in further calculations. Other spectral regions include a region between 1.05 to 1.4 ppm, which is primarily alkane methylene, although beta methyl and gamma methylene protons may also be present here. The region between 1.4 and 2.0 ppm, contains alkane methine, cyclo-alkane methylene, as well as beta methylene protons. All alpha protons occur

**TABLE 6. NMR Chemical Shift Correlations
(Bailey's Method)**

<u>Proton Assignments</u>	<u>Shift Range, ppm from TMS</u>
Alkane and gamma* methyl	0.5 - 1.05
Alkane methylene, beta methyl, gamma methylene	1.05 - 1.4
Alkane methine, cycloalkane methylene, beta methylene	1.4 - 2.0
Alpha methyl, methylene, methine	2.0 - 4.4
Alpha methyl	2.0 - 2.3
Aromatic protons	6.2 - 9.2
<u>Carbon Assignments</u>	
Aromatic carbon	118.0 - 160.0
Protonated carbon	115.0 - 130.5
Saturated carbon and gamma methyl	14.2
* Alpha, beta, and gamma indicate locations relative to nearest aromatic ring.	

**TABLE 7. Average Molecular Parameter Calculations
(Bailey's Method)**

Average molecular weight = (UOP Method + API Method)/2	EQ. 1
Aromatic H (¹³ C) = MW(C,wt%)(¹³ C integral, 118.0-130.5 ppm)/120,000	EQ. 2
Aromatic H (¹ H) = MW(H,wt%)(¹ H integral, 6.2-9.2 ppm)/10,000	EQ. 3
Gamma Methyl C (¹³ C) = MW(C,wt%)(¹³ C integral, 14.2 ppm)/120,000	EQ. 4
Gamma Methyl C (¹ H) = MW(H,wt%)(¹ H integral, 0.5-1.05 ppm)/10,000	EQ. 5
Aromatic C (¹³ C) = MW(C,wt%)(¹³ C integral, 118.0-160.0 ppm)/120,000	EQ. 6
Percent unsubstituted carbon (¹³ C) = (EQ. 2)/(EQ. 6)	EQ. 7
Percent unsubstituted carbon (¹ H) = (EQ. 3)/(EQ. 6)	EQ. 8
H/C atomic ratio, alkyl groups = (100- ¹ H integral, 6.2-9.2 ppm)(H,wt%)/ (100- ¹³ C integral, 118.0-160.0 ppm)(C,wt%)/12	

**TABLE 8. Nuclear Magnetic Resonance Analysis
(Bailey's Method)**

¹ H and ¹³ C Identification	Chemical Shift, ppm	Fuel Identification			
		16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
C (aromatic)	118.0 - 160.0	16.5	19.8	24.2	40.0
C (ar, protonated)	115.0 - 130.5	9.8	12.0	14.7	24.9
C (sat. gamma methyl)	14.2	16.4	22.3	13.5	8.3
H (aromatic)	6.2 - 9.2	5.6	7.8	8.9	16.3
H (sat. gamma methyl)	0.5 - 1.05	32.6	32.1	19.2	18.7
H (sat. methylene, beta methyl, gamma methylene)	1.05- 1.4	49.3	48.2	48.2	37.4
H (sat. methine, beta and cycloalkyl methylene)	1.4 - 2.0	7.9	6.0	11.2	7.9
H (alpha methyl, methylene and methine)*	2.0 - 4.4	4.7	6.0	12.5	19.7
H (alpha methyl)*	2.0 - 2.3	2.3	2.3	7.1	6.9

* With respect to an aromatic ring.

between 2.0 and 4.4 ppm, but the alpha methyl protons may be separated out in the 2.0- to 2.3-ppm range. Some basic observations may be made from the integral percentage measurements presented in TABLE 8. It is interesting to note that all four fuels have very similar molecular weights, yet the structural information from NMR shows definite differences. The quantity of aromatic hydrogen varies from 5.6 to 16.3 percent. These figures do not give an unambiguous indication of the fuels' aromatic hydrocarbon content, unless the degree of substitution is known. Aromatic carbon, as measured by ¹³C NMR, is a stronger and more direct indicator of fuel aromaticity. This may be considered the most reliable NMR measurement of the aromatic character of a fuel. Because these samples were not separated into aromatic and saturate fractions for NMR studies, the structures of alkyl substituents on the aromatic rings are in doubt. Some estimates of the degree of aromatic alkyl substitution and quantity of terminal chain carbons can be made. These estimates can be normalized by basing the calculations on the average molecular structure.

TABLE 9 contains some calculated average molecular parameters derived from the basic NMR quantitative data by Bailey's method, the average molecular weights and results of carbon and hydrogen elemental analysis. To compare the two NMR analyses (i.e., ^1H and

TABLE 9. Calculated Average Molecular Parameters
(Bailey's Method)

Molecular Parameters	Fuel Identification			
	16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
Comparison of ^{13}C and ^1H NMR Data				
Aromatic protons (^{13}C)	1.6	2.0	2.2	5.6
Aromatic protons (^1H)	1.5	1.7	2.2	3.9
Gamma methyl carbons (^{13}C)	2.5	3.2	2.0	1.3
Gamma methyl carbons (^1H)	3.0	2.8	1.6	1.4
Total Aromatic Carbon				
Aromatic carbons (^{13}C)	2.6	2.9	3.6	6.3
Percent unsubstituted (^{13}C)	57.7	58.6	61.1	61.9
Percent unsubstituted (^1H)	61.5	69.0	61.1	88.9
HPLC polars (for comp.) wt%	25.7	33.0	44.5	65.3
Hydrogen-to-Carbon Atomic Ratio				
Alkyl groups (^1H and ^{13}C)	2.04	2.05	2.02	2.01

^{13}C - NMR), the number of protonated aromatic carbons were calculated from both proton and ^{13}C data. Three of the model fuels showed good agreement for this parameter. A small discrepancy was noted for the LCO, for which the proton NMR data indicated a larger proportion of protonated aromatic carbons. Gamma methyl carbons per average molecule were also calculated from both spectra with fairly good agreement. From the ^{13}C spectra, the total number of aromatic carbons per average molecule were calculated. Again, the spread in aromaticity between the four fuels is demonstrated by this parameter, and the general order of aromatic character was maintained. The weight percent of polar compounds, as measured by HPLC, is also included in the table for comparison. These results are quite consistent with the calculated aromatic carbon parameter. The percentages of aromatic carbons that are unsubstituted were also calculated from the two NMR spectra with fairly consistent results, showing about 60 percent of the aromatic carbon being found in an unsubstituted

state. One exception to this calculation was made from proton NMR data. This showed that nearly 90 percent of the aromatic carbon is in the unsubstituted state for the light cycle oil. This parameter calculated from the ^{13}C data showed only 62 percent unsubstituted carbon. The calculated carbon-to-hydrogen atomic ratio for the alkyl groups is near the expected two hydrogen atoms for each carbon atom.

C. GC/MS Analysis of Model Fuels

High-resolution gas chromatography using mass spectroscopic detection (GC/MS) yields definitive compositional data. Each of the three diesel fuels and the light cycle oil were analyzed by this method. To ease interpretation of the GC/MS results, the complexity of each of the products was reduced by preseparating them into two fractions, consisting of (a) nonpolar compounds, mainly saturated hydrocarbons, and (b) polar compounds, principally aromatic hydrocarbons. These preseparations were performed using high-performance liquid chromatography (HPLC) with activated silica gel columns. Elution of the saturated fraction was accomplished using n-hexane, while the polar compounds were "back-washed" with methylene chloride. The solvents were evaporated at room temperature under vacuum. The separated fractions were then individually analyzed by high resolution capillary GC/MS.

Gas chromatographic conditions are shown in TABLE 10. The capillary column of the GC was directly interfaced into the mass spectrometer's source through a heated interface tube held at 280°C . The operating conditions for the mass spectrometer are summarized in TABLE 11.

The mass spectrometer-generated total ion chromatograms (TIC) were integrated at a threshold level that projected the baseline horizontally from start to the end. This action caused all peak areas to be integrated down to the baseline, thus allowing for the quantitation of the entire sample, including both resolved and unresolved components.

This total integration listed between 300 to 500 peaks, depending on the sample. The integration threshold level was then adjusted to detect only those peaks that the total integration showed to be approximately 0.2 percent or greater. This adjustment yielded integrated TICs of about 100 peaks. The mass spectrum of each of these peaks was generated and subjected to computerized library search using the Wiley Library data

TABLE 10. Gas Chromatographic Conditions for the Analysis of Fuel Fractions

Gas Chromatograph:	Hewlett-Packard Model 5890
Injector Temperature:	300°C
Injector Split Ratio:	100:1, Helium
Injection Volume:	1 microliter
Column:	50 m x 0.2 mm ID SE-54
Oven Temperature:	Programmed
Initial Temperature:	0°C
Initial Hold:	0.00 min.
Program Rate:	3.0°C/min.
Final Temperature:	320°C
Final Hold:	10 min.
Flow Rate at 150°C:	0.65 mL He/min.

TABLE 11. Mass Spectrometer Operating Conditions

Solvent Delay:	0.00 min.
eV Volts:	0 Relative
Resulting Voltage:	1800
Start Time:	0.00 min.
Low Mass Limit:	10 m/z
High Mass Limit:	550 m/z
Scan Threshold:	20 counts
a/d Samples (2 N):	2
Scans/Second:	0.79

base, containing about 80,000 spectra, and the NBS Library, containing more than 38,000 spectra. The generated spectrum and library search results were then studied to confirm or correct identifications and quantitative values assigned based on the results of the total integration of the TIC. The detailed GC/MS data are tabulated in Appendix A-1. Reconstructed total ion chromatograms (TIC) for the model fuel fractions are

TABLE 12. GC/MS Analysis of the Middle Distillate Products

Class of Compounds	Product Identification Number			
	16058 (Cat)	15978 (EPA)	16082 (Ref)	15980 (LCO)
Nonaromatic Fraction:				
normal-paraffins	24.62	20.71	14.71	11.95
iso-paraffins	21.88	25.32	25.72	14.16
cyclo-paraffins	4.32	2.22	0.28	0.53
olefins	ND*	0.20	0.18	ND
Total identified by				
GC/MS, wt%	50.82	48.45	41.89	26.64
HPLC (100% baseline), wt%	74.3	67.0	55.5	34.7
100(GC/MS)/HPLC, wt%	68.4	72.3	75.5	76.8
Aromatic Fraction:				
benzenes	1.75	3.70	8.63	1.97
tetralins	0.38	1.65	2.75	0.30
naphthalenes	6.75	9.64	5.11	18.82
indenes and indanes	ND	ND	0.63	0.43
biphenyls	0.51	0.28	0.30	4.34
biphenylenes	0.41	ND	ND	ND
fluorenes	1.08	0.12	0.23	1.78
anthracenes	ND	0.21	ND	2.13
phenanthrenes	1.52	0.64	ND	9.00
pyrenes	ND	ND	ND	0.21
fluoranthenes	ND	ND	ND	1.09
azulenes	0.40	0.13	ND	0.40
phenols	0.19	ND	ND	ND
dibenzofuranes	0.27	1.12	ND	ND
dibenzothiophenes	0.27	0.78	0.07	1.09
carbazoles	ND	ND	ND	0.30
Total identified by				
GC/MS, wt%	13.59	18.27	17.72	41.86
HPLC (100% baseline), wt%	25.7	33.0	44.5	65.3
100(GC/MS)/HPLC, wt%	52.9	55.4	39.8	64.1

* ND = Not Detected.

shown in Appendix A-2. Results of the GC/MS analysis are summarized according to compound classes are given TABLE 12.

Due to the restricted integrated threshold levels set for the various peaks, substantially less than 100 percent of each fuel fraction was identified. The gravimetrically measured HPLC data were defined as "100 percent" for each of the two fractions from each fuel. The GC/MS data were then compared to this HPLC-derived baseline. As

shown in TABLE 12, identification of the nonaromatic fraction amounted to between 68 and 77 wt%, while identification of the aromatic fraction varied between 40 and 55 wt%.

VI. FUEL-ELASTOMER COMPATIBILITY STUDIES

The ultimate test for the compatibility of a storage tank with a stored material is through mutual long-term exposure, in which neither material contaminates, nor otherwise deteriorates the other. Such exposures are usually not feasible. Laboratory simulation of the compatibility of collapsible elastomeric fuel tanks with the various fuels have been only partially addressed in specifications. At present, no specification exists for the evaluation of middle distillate fuels in such tanks. Since a number of fuels may need to be stored in a variety of elastomeric fuel tanks, it is imperative that their compatibility be established by the proper selection of tank materials and that these materials be exposed to either (a) a well chosen fuel (or a combination of fuels), or (b) that these materials be exposed to correctly designed mixture(s) of pure "model" compounds. The first method would thus use "realistic" fluid(s), while the second would expose the elastomers to repeatable, constant composition fluid(s). It is also a project objective to resolve this issue.

In the present study, the four model fuels were exposed to four elastomers having varied, but undisclosed, hydrolytic stability and fuel resistance. It was expected that preoxidized (aged) fuels might cause enhanced elastomer degradation because the increased concentrations of polar oxidation products are more likely to attack the polar elastomers. For this reason, not only the fresh but also the preoxidized fuels were exposed to the same elastomers. The effects of the fuels on the elastomers were to be studied by U.S. Army Belvoir Research, Development and Engineering Center (Belvoir RDE Center), while the effects of the elastomers on the fuels were to be analyzed by Belvoir Fuels and Lubricants Research Facility (SwRI) (BFLRF).

A series of experiments was carried out to determine the conditions for fuel preoxidation. Each of the four procured products was oxidized for various stress periods and temperatures in ASTM D 2274 equipment. To follow the extent of product aging, measurements of the quantity of precipitate, adherent gum, steam jet gum (SJG), peroxide number (PN), and color were made as a function of stress time at 100° and

120°C. To further follow the extent of oxidation, infrared and differential infrared spectra were generated using the unaged and aged fuels. Results of this work are summarized in TABLE 13. It was concluded that the light cycle oil should be preaged at 100°C in 24 hours, while the three diesel fuels should be preoxidized at 120°C in 72 hours. Accordingly, a quantity of each of the fuels was preaged to satisfy the immediate needs of both Belvoir RDE Center and BFLRF. The unaged and preaged samples were sparged with argon to alleviate further oxidation reactions. Aliquots of these samples were shipped to Belvoir RDE Center, while the remaining samples were retained at BFLRF.

Evaluation of the elastomers on the unaged and aged fuels followed a prearranged protocol. The elastomers, designated as samples A, B, C, and D, were cut into 1- to 2-millimeter samples. Fifteen grams of the cut elastomer samples were measured into 1-liter capacity borosilicate bottles, each containing 750 mL of the appropriate fuel. Screw-on caps covered the flasks, allowing minor "breathing" of the contents. The samples were placed into an oil bath in which the temperature was held at $80 \pm 1^\circ\text{C}$ for a 14-day period. To improve the diffusion-controlled process of elastomer-fuel interactions, the flasks were mildly agitated daily. After the 14-day exposure, the fuel samples were decanted from the elastomer samples to allow partial analysis of the exposed fuels. The results of these analyses are tabulated in TABLE 14.

The changes that took place in the fuels due to (a) preaging, (b) exposure to 80°C for 14 days, and (c) exposure to 80°C in the presence of the various elastomers, were monitored by measurements of color, steam jet gum contents, peroxide concentrations, flash points, total acid numbers, and JFTOT ratings at 279°C. Additionally, IR and differential IR were obtained on each of the middle distillates before and after the exposures at elevated temperatures.

Analysis of the infrared spectra only revealed the presence of the saturated and aromatic hydrocarbons, and that the concentration of the aromatic hydrocarbons varied from fuel to fuel. Differential infrared spectra that were generated by the comparison of aged and unaged fuels were essentially featureless. Similarly, the possible effects of the elastomers on either the unaged or aged fuel samples could not be resolved by these techniques. These observations do not rule out interactions among fuel and elastomer components, as the concentrations of products may be below the detectability limits of the applied IR methods.

TABLE 13. Determination of Conditions for Preoxidation

Fuel No.	Modified ASTM D 2274					SJG, mg/100 mL	PN, ppm	Color
	Temp., °C	Hours	Prec.	Adher.	Total			
16058 (Cat)	100	0	--	--	--	4.2	1.6	1.0
	100	21	2.2	0.5	2.7	8.6	3.6	L 3.0
	100	45	4.2	1.4	5.6	13.3	4.3	4.0
	100	69	5.8	2.5	8.3	24.5	5.1	4.5
	100	93	6.3	3.2	9.5	22.3	4.0	L 5.5
15978 (EPA)	100	0	--	--	--	5.6	0.0	L 2.5
	100	21	0.1	0.1	0.2	6.7	0.0	--
	100	45	0.2	0.1	0.3	8.3	1.0	--
	100	69	0.2	0.3	0.5	9.4	2.0	--
	100	93	0.4	0.3	0.7	10.3	2.1	--
16082 (Ref)	100	0	--	--	--	3.5	1.3	L 1.0
	100	21	2.5	0.5	3.0	67.3	25.3	4.5
	100	45	15.8	3.3	19.1	84.0	28.5	6.0
	100*	69	25.8	8.2	34.0	155.1	35.4	7.0
	100*	93	36.1	9.7	45.8	164.8	25.6	L 7.5
15980 (LCO)	100	0	--	--	--	6.7	2.0	2.0
	100	2	0.5	0.1	0.6	14.0	1.8	2.5
	100	4	0.3	0.1	0.4	18.2	3.4	2.5
	100	6	0.8	0.0	0.8	24.0	4.5	3.0
	100	8	0.3	0.1	0.4	25.2	5.9	3.5
	100	24	2.7	0.5	3.2	108.5	25.7	L 7.5
16058 (Cat)	120	0	--	--	--	4.2	1.6	1.0
	120	21	7.2	2.5	9.7	13.9	2.8	4.5
	120	45	14.6	3.8	18.4	36.4	7.2	7.0
	120	69	24.2	7.2	31.4	61.5	12.8	7.5
15978 (EPA)	120	0	--	--	--	5.6	0.0	L 2.5
	120	21	0.4	3.7	4.1	10.7	2.4	7.5
	120	45	2.3	0.1	2.4	12.0	7.1	7.5
	120	69	6.1	2.4	8.5	17.0	15.6	8.0
16082 (Ref)	120	0	--	--	--	3.5	1.3	L 1.0
	120	21	10.9	19.4	30.3	102.5	13.3	7.5
	120	45	57.0	15.5	72.5	172.8	17.5	D 8.0
	120	69	84.9	27.2	112.1	278.4	30.0	D 8.0

Notes: SJG = Steam jet gum.

PN = Peroxide number.

L = Color lighter than indicated, D = Color darker than eight (8).

* Filter plugged in 2 hours.

**TABLE 14. Evaluation of the Effects of Elastomers on
Unaged and Aged Fuels**

Ref No.	Fuel I.D.	Fuel, mL	Elastomer		Color*	After Exposure at 80°C for 14 Days						
			I.D.	Wt, G		SJG, mg/dL	PN, ppm	Flash Pt., °C	TAN*	JFTOT at 279°C		
										Vis.**	TDR*	ΔP**
1	16058 Cat	New Fuel Blank	--	--	1.0	4.5	1.6	86	0.03	4	26/29	-0-
2		750	-	--	L 4.0	8.7	4.2	86	0.03	4	25/28	-0-
3		750	A	15	L 3.0	16.5	2.5	86	0.02	3	21/31	-0-
4		750	B	15	L 3.0	8.7	3.7	86	0.03	4	25/26	-0-
5		750	C	15	L 3.0	34.4	3.2	86	0.03	2	22/23	-0-
6		750	D	15	L 3.0	22.0	4.1	86	0.03	3	23/25	-0-
7	16058 Aged Cat	Aged Fuel Blank	--	--	8.0	56.2	10.0	102	0.11	>4	26/28	125/63
8		750	-	--	8.0	53.7	10.1	99	0.10	>4B	25/29	125/147
9		750	A	15	7.5	52.0	9.6	99	0.08	>4	24/25	-0-
10		750	B	15	7.5	61.5	13.3	101	0.03	>4B	27/29	-0-
11		750	C	15	7.5	69.4	14.5	99	0.03	>4	17/18	-0-
12		750	D	15	L 8.0	52.3	13.7	101	0.02	>4	16/17	35/150
13	15978 EPA	New Fuel Blank	--	--	L 2.5	5.9	0.0	64	0.02	2	13/15	-0-
14		750	-	--	6.5	26.9	4.8	69	0.03	2	14/16	-0-
15		750	A	15	L 3.0	21.2	0.0	68	0.02	3	26/27	-0-
16		750	B	15	L 3.0	10.0	4.9	69	0.01	3	25/28	-0-
17		750	C	15	L 3.0	25.0	1.8	68	0.02	4	25/28	-0-
18		750	D	15	L 3.0	8.3	0.0	68	0.02	4G	23/24	-0-
19	15978 Aged EPA	Aged Fuel Blank	--	--	L 6.0	18.1	6.0	83	0.03	3	24/25	-0-
20		750	-	--	5.0	18.1	7.8	83	0.02	4	28/36	-0-
21		750	A	15	L 4.5	28.3	3.2	83	0.02	<3	20/23	-0-
22		750	B	15	L 5.0	17.9	6.3	83	0.02	>3	29/33	-0-
23		750	C	15	4.5	29.1	4.5	83	0.02	4	26/27	-0-
24		750	D	15	L 5.0	17.9	3.8	81	0.02	>4	32/36	-0-
25	16082 Ref	New Fuel Blank	--	--	L 1.0	3.0	1.3	57	0.03	3	20/22	-0-
26		750	-	--	4.0	27.5	16.1	59	0.04	4	26/29	-0-
27		750	A	15	L 3.5	32.7	16.0	61	0.00	<4	20/23	-0-
28		750	B	15	3.0	20.0	16.3	59	0.00	>4	24/26	116/150
29		750	C	15	2.5	29.2	11.4	61	0.01	>4	20/27	125/78
30		750	D	15	3.5	27.7	18.9	61	0.00	>4	21/24	125/139
31	16082 Aged Ref	Aged Fuel Blank	--	--	8.0	291.3	22.6	88	0.33	>4B	26/28	125/85
32		750	-	--	D 8.0	188.9	26.7	85	0.15	>4	23/27	-0-
33		750	A	15	8.0	185.3	26.0	86	0.15	PC	33/35	125/122
34		750	B	15	D 8.0	153.0	27.0	88	0.15	PC	31/32	125/103
35		750	C	15	8.0	182.4	28.6	86	0.17	>4G	21/26	-0-
36		750	D	15	8.0	167.4	25.0	88	0.11	PC	27/24	125/127
37	15980 LCO	New Fuel Blank	--	--	2.0	7.1	2.0	112	0.03	3	21/27	-0-
38		750	-	--	D 8.0	188.7	23.0	109	0.02	<3	22/31	-0-
39		750	A	15	D 8.0	124.8	19.4	111	0.02	<4	43/46	-0-
40		750	B	15	8.0	114.1	31.9	113	0.02	<3P	19/22	-0-
41		750	C	15	6.5	94.6	16.8	113	0.02	<4	37/42	-0-
42		750	D	15	L 8.0	84.4	17.3	113	0.02	<4	30/36	-0-
43	15980 Aged LCO	Aged Fuel Blank	--	--	L 7.5	91.5	24.0	116	0.03	<4	31/32	-0-
44		750	-	--	D 8.0	211.4	31.0	116	0.04	2	10/13	-0-
45		750	A	15	D 8.0	153.5	23.5	113	0.04	2	20/37	-0-
46		750	B	15	D 8.0	143.0	22.8	116	0.03	3	28/34	-0-
47		750	C	15	D 8.0	168.0	24.1	113	0.02	<3	20/26	-0-
48		750	D	15	D 8.0	162.0	25.8	113	0.04	<4	25/29	-0-

Notes: SJG = Steam Jet gum.
 PN = Peroxide number.
 L = Color lighter than indicated, D = Color darker than eight (8).
 * ASTM D 664, Using 0.01N KOH. Units mg KOH/g Fuel.
 ** B = Gun-Metal Blue, G = Green, PC = Peacock, and P = Equipment Problem.
 * TDR Spun/Spot Deposit Ratings.
 ** ΔP, mm Hg/Minutes.

The experimental results are tabulated in TABLE 14, while JFTOT evaluations by the spun tube rating (TDR) measurements for all fuel-elastomer-aging combinations are also graphically illustrated in Fig. 2. The data do not yield clear trends in fuel-elastomer compatibility. While it is expected that extended high-temperature exposure of fuels should result in darkening of the colors, increasing gum contents, peroxides, and

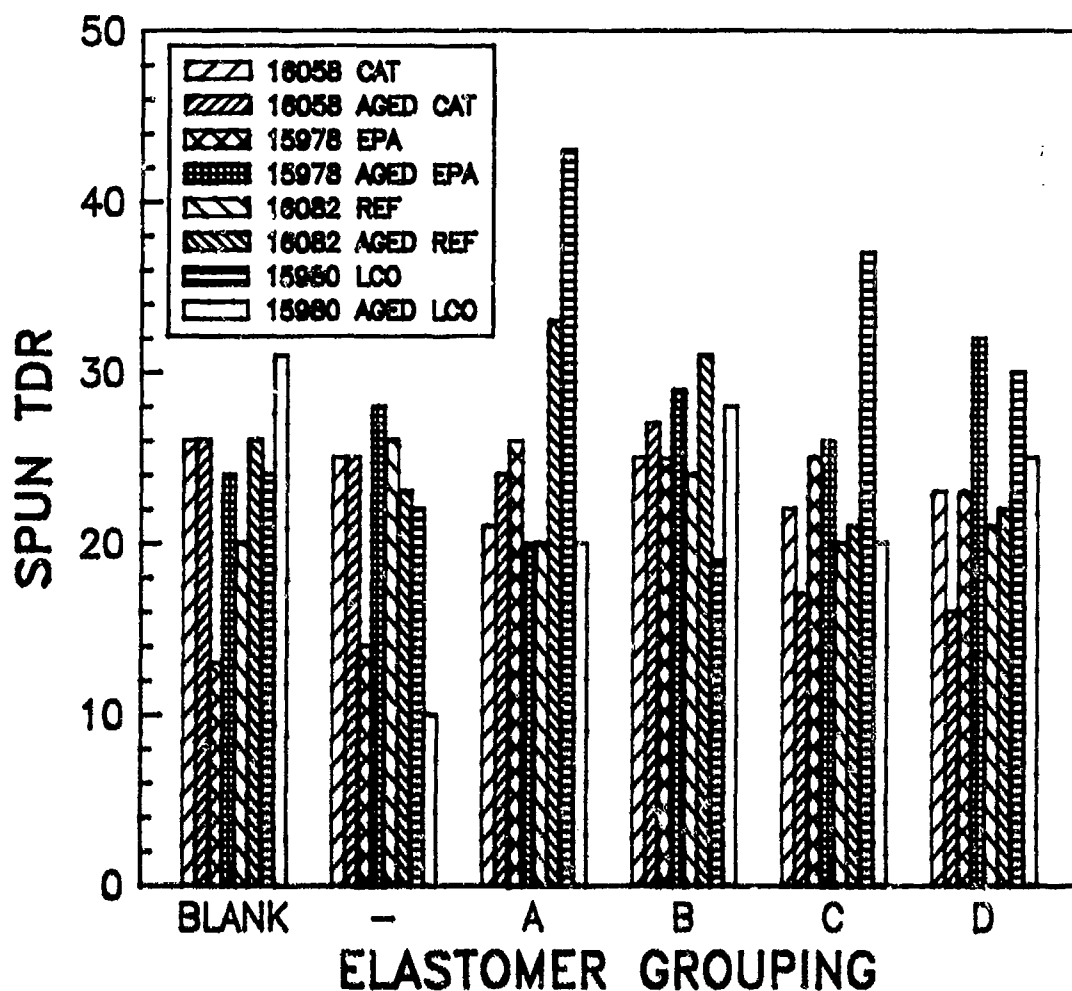


Figure 2. Spun TDR ratings for all fuel-elastomer-aging combinations

acidic components, and degradation of the results of JFTOT evaluations, none of these expectations were unambiguously fulfilled. Although color bodies in the fuels normally increased with increased exposure, reversals of this trend were also found. A possible explanation of this observation may be that some of the formed color bodies adsorbed on the surface or absorbed into the elastomers. This reasoning is supported by the observation that all the fuel-exposed elastomers became dark.

More pronounced effects may also have taken place. While attempting to dissolve the color bodies from the elastomer surfaces, some variations were found among the polymers. It was found that none of these materials was heptane soluble, and that, upon heptane wash, the color and the apparent consistency of the elastomers remained unchanged. When these gum-contaminated elastomers were washed with an equivolume mixture of toluene, acetone, and methanol (TAM), Elastomer D from Experiment Nos. 12, 30, and 36 essentially disintegrated when touched by a spatula. Elastomer C was similarly affected in Experiment No. 35, and Elastomer B was somewhat affected by the same treatment in Experiment No. 34. At this time, no explanation may be offered for these observations.

Gum content of the stressed fuels also show interesting variations. After exposure of the unaged EPA fuel (No. 15978) to Elastomers B and D at 80°C for 14 days, the gum content of the decanted fuel was substantially less than that of the elastomer-free fuel. Similarly, the unaged LCO (No. 15980) produced reduced gum content in the presence of Elastomers C and D. This observation may also be explained by adsorption of gum components on the surface of these elastomers; thus, only reduced gum concentrations were available in the decanted fuel samples.

Peroxide concentrations in the decanted fuels do not show strong trends that may be due to the presence of the elastomers.

Flash point measurements indicate that during the initial preoxidation (preaging) of the diesel fuels some of the low boiling point components evaporated from the reaction tubes described in ASTM D 2274. This loss, which amounted to about 3.0 to 3.5 vol% of the fuel, was obviously due to insufficient capacity of the reflux condenser under the modified ASTM D 2274 conditions.

Acid number measurements showed somewhat unexpected results only in case of the unaged referee grade diesel fuel (No. 16082). In this case, the elastomer-exposed fuel samples gave lower acid number values than the blank sample.

Evaluations of the effects of exposure of unaged and aged fuels to the various elastomers by the JFTOT method was not possible, as no differentiation could be noted among the samples of the experimental matrix.

VII. CONCLUSIONS AND RECOMMENDATIONS

It may be concluded that evaluation of fuel-elastomer interactions requires application of different methodologies. It seems apparent that whatever effects the elastomers may have had on the fuels, these effects were masked by the overwhelming effects of fuel aging. Inclusion of new methodologies may include the study of the effects of exposure of elastomers to fuels under inert, i.e., non-oxidizing environments, or replacement of "real" fuels with an appropriately selected mixture of model compounds. Since the composition of such a fuel simulant is appreciably simpler than that of a full boiling range fuel, reactions that may take place between the model compounds and elastomers may be better controlled, and the results better understood.

The analytical results that describe the four fuels' composition, while not giving full account of the fuels, give strong indications of compounds actually present in the examined products. Such methodology may be improved by, for example, the increased use of reference materials. It is expected that extension of such analyses to a number of additional diesel fuels will lead to a compositional data base that may be used to develop an "average" No. 2 diesel fuel composition. This information, in turn, may be used to design a repeatable quality model compound based diesel fuel simulant. (It may be necessary to develop other fuel simulant compositions for other middle distillate products.) Additionally, these fuel compositional data should be useful in explaining and ultimately governing fuel-elastomer interactions.

It is fully expected that ultraviolet radiation strongly contributes to the degradation of both fuels and elastomeric fuel tanks. The effects of even a partially degraded fuel tank material may be profound on the fuel that it may contain. It would be of advantage, therefore, to study and understand the effects and interactions caused by ultraviolet radiation on fuels, elastomer-based fuel tanks, and their combinations.

VIII. LIST OF REFERENCES

1. Letter, STRBE-VF, dated 19 February 1987.
2. Letter, STRBE-VF, dated 27 March 1987.
3. Letter, BFLRF(SwRI), dated 12 March 1987.
4. Memorandum for Record, STRBE-VF, dated 23 March 1987.
5. Infomail STRBE-VF to BFLRF (SwRI), dated 8 April 1987.
6. Letter, Input on Test Fuel Requirements, BFLRF (SwRI), dated 02 December 1986.
7. Project Plan on Elastomer Test Fluid Development, BFLRF (SwRI), dated 24 April 1987.
8. Shertzer, R.H., "Organic Peroxides in JP-5 Investigations," Final Report, NAPC-433, 1978.
9. Hazlett, R.N., Hall, J.M., Nowack, C.J., Craig, L., "Hydroperoxide Formation in Jet Fuels," Proc. of Conference on Long-Term Stabilities of Liquid Fuels, Paper No. B132, Tel Aviv, Israel, 11-14 July 1983.
10. Russell, J.A., et al., "Development of Accelerated Fuel-Engine Qualification Procedure Methodology," Interim Report AFLRL No. 144, AD A113461, December 1981.
11. Russell, J.A., Cuellar, Jr., J.P., Alvarez, R.A., Rentz, R.L., and Timbario, T.J., "Development of Accelerated Fuel-Engine Qualification Procedures," Final Technical Report No. SwRI-6794/4, 30 September 1983.
12. Gillet, S., Rubini, P., Delpuech, J.J., Escalier, J.C., and Valentin, P., "Quantitative Carbon-13 and Proton Nuclear Magnetic Resonance Spectroscopy of Crude Oil and Petroleum Products," Parts I and II, Fuel, Vol 60, pp. 221-230, 1981.
13. Dalling, D.K., Bailey, B.K., and Pugmire, R.J., "Carbon-13 and Proton Nuclear Magnetic Resonance Analysis of Shale-Derived Refinery Products and Jet Fuels and of ERBS Jet Fuels," NASA Report 174761, September 1984.
14. Bailey, B.K., et al., "Cetane Number Prediction from Proton-Type Distribution and Relative Hydrogen Population," SAE 861521, October 1986.
15. Fodor, G.E. and Newman, F.M., "The Application of High-Performance Liquid Chromatography to the Analysis of Petroleum Materials. Part 1. Qualitative Hydrocarbon-Type Analysis," Interim Report AFLRL No. 62, AD A014994, May 1975; and Part 2, "Quantitative Hydrocarbon-Type Analysis", Interim Report AFLRL No. 63, AD A014996, May 1975.

APPENDIX
Detailed GC/MS Data

APPENDIX A-1
Detailed GC/MS Values

POLAR FRACTION OF FUEL NO. 16058

Analysis Summary

C ₅ - substituted benzenes	-	0.03%		
C ₆ - substituted benzenes	-	0.05%		
c-C ₆ - substituted benzenes	-	0.15%		
C ₇ - substituted benzenes	-	0.05%		
C ₈ - substituted benzenes	-	0.21%		
C ₉ - substituted benzenes	-	0.33%		
C ₁₀ - substituted benzenes	-	0.24%		
C ₁₁ - substituted benzenes	-	0.33%		
C ₁₂ - substituted benzenes	-	0.18%		
C ₁₃ - substituted benzenes	-	0.12%		
C ₁₅ - substituted benzenes	-	0.06%	Subtotal	1.75%
tetralin	-	0.04%		
C ₂ - substituted tetralins	-	0.17%		
C ₃ - substituted tetralins	-	0.17%	Subtotal	0.38%
naphthalene	-	0.08%		
C ₂ - substituted naphthalenes	-	0.57%		
C ₃ - substituted naphthalenes	-	1.52%		
C ₄ - substituted naphthalenes	-	1.74%		
C ₅ - substituted naphthalenes	-	0.63%		
C ₆ - substituted naphthalenes	-	2.05%		
C ₇ - substituted naphthalenes	-	0.16%	Subtotal	6.75%
biphenyl	-	0.12%		
C ₁ - substituted biphenyls	-	0.24%		
C ₃ - substituted biphenyls	-	0.15%	Subtotal	0.51%
C ₆ - substituted azulenes	-	0.31%		
C ₇ - substituted azulenes	-	0.15%	Subtotal	0.46%
C ₃ - substituted fluorene	-	0.13%		
C ₄ - substituted fluorene	-	0.15%		
C ₅ - substituted fluorene	-	0.36%		
C ₆ - substituted fluorene	-	0.44%	Subtotal	1.08%
C ₆ - substituted biphenylene	-	0.41%	Subtotal	0.41%
C ₇ - substituted phenol	-	0.19%	Subtotal	0.19%
C ₄ - substituted dibenzofurans	-	0.27%	Subtotal	0.27%
C ₃ - substituted phenanthrenes	-	0.29%		
C ₆ - substituted phenanthrenes	-	0.71%		
C ₇ - substituted phenanthrenes	-	0.52%	Subtotal	1.52%
C ₄ - substituted dibenzothiophenes	-	0.16%		
C ₅ - substituted dibenzothiophenes	-	0.11%	Subtotal	0.27%
			TOTAL	13.59%

POLAR FRACTION OF FUEL NO. 16058 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
38.282	C ₅ - substituted benzene	0.12	0.03
38.795	tetralin	0.14	0.04
40.012	naphthalene	0.32	0.08
43.367	C ₂ - substituted naphthalene	0.14	0.04
43.842	C ₆ - substituted benzene	0.18	0.05
44.229	C ₂ - substituted tetralin	0.46	0.12
45.856	C ₂ - substituted naphthalene	2.06	0.53
46.094	C ₂ - substituted tetralin	0.20	0.05
46.679	C ₃ - substituted naphthalene	1.01	0.26
46.941	c-C ₆ - substituted benzene	0.59	0.15
47.854	C ₃ - substituted tetralin	0.29	0.07
48.116	C ₃ - substituted tetralin	0.16	0.04
48.377	C ₇ - substituted benzene	0.21	0.05
48.724	C ₃ - substituted tetralin	0.23	0.06
49.772	biphenyl	0.45	0.12
50.448	C ₃ - substituted naphthalene	0.47	0.12
50.513	C ₃ - substituted naphthalene	0.38	0.10
50.668	C ₃ - substituted naphthalene	0.34	0.09
51.177	C ₃ - substituted naphthalene	2.42	0.62
51.922	C ₄ - substituted naphthalene	2.49	0.64
52.078	C ₃ - substituted naphthalene	1.28	0.33
52.166	C ₄ - substituted naphthalene	0.28	0.07
52.715	C ₄ - substituted naphthalene	0.90	0.23
52.824	C ₄ - substituted naphthalene	0.58	0.15
53.071	C ₈ - substituted benzene	0.53	0.14
53.334	C ₄ - substituted naphthalene	0.59	0.15
53.898	C ₈ - substituted benzene	0.29	0.07
54.607	C ₁ - substituted biphenyl	0.94	0.24
54.783	C ₄ - substituted naphthalene	0.48	0.12
55.075	C ₉ - substituted benzene	1.01	0.26
55.517	C ₄ - substituted naphthalene	1.10	0.28
55.718	C ₄ - substituted naphthalene	0.37	0.10
55.850	C ₉ - substituted benzene	0.28	0.07
55.983	C ₅ - substituted naphthalene	0.52	0.13
56.228	C ₅ - substituted naphthalene	0.84	0.22
56.494	C ₆ - substituted naphthalene	1.12	0.29
56.742	C ₆ - substituted naphthalene	1.21	0.31
56.988	C ₅ - substituted naphthalene	0.61	0.16
57.387	C ₆ - substituted naphthalene	0.81	0.21
57.542	C ₆ - substituted naphthalene	1.21	0.31
58.118	C ₆ - substituted azulene	1.35	0.35
58.231	C ₆ - substituted naphthalene	0.50	0.13
58.837	C ₆ - substituted naphthalene	0.58	0.15
58.926	C ₃ - substituted fluorene	0.50	0.13
59.104	C ₆ - substituted naphthalene	0.86	0.22
59.461	C ₆ - substituted naphthalene	0.54	0.14
59.550	C ₃ - substituted biphenyl	0.57	0.15

POLAR FRACTION OF FUEL NO. 16058 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
59.841	C ₅ - substituted naphthalene	0.45	0.12
60.211	C ₆ - substituted naphthalene	0.74	0.19
60.435	C ₆ - substituted naphthalene	0.63	0.16
60.571	C ₆ - substituted naphthalene	0.39	0.10
60.999	C ₄ - substituted dibenzofuran	0.74	0.19
61.447	C ₇ - substituted dibenzofuran	0.33	0.08
61.536	C ₆ - substituted azulene	0.47	0.12
61.672	C ₁₀ - substituted benzene	0.58	0.15
62.298	C ₁₀ - substituted benzene	0.36	0.09
62.410	C ₆ - substituted azulene	0.73	0.19
63.065	C ₇ - substituted azulene	0.59	0.15
63.177	C ₇ - substituted naphthalene	0.61	0.16
63.446	C ₁₁ - substituted benzene	0.63	0.16
63.581	C ₄ - substituted fluorene	0.60	0.15
63.897	C ₅ - substituted fluorene	1.40	0.36
64.939	C ₆ - substituted biphenylene	1.10	0.28
65.118	C ₆ - substituted biphenylene	0.52	0.13
65.658	C ₇ - substituted phenol	0.75	0.19
65.726	C ₆ - substituted fluorene	0.37	0.10
66.268	C ₁₁ - substituted benzene	0.68	0.17
66.770	C ₃ - substituted phenanthrene	1.12	0.29
67.946	C ₆ - substituted fluorene	0.65	0.17
68.127	C ₆ - substituted fluorene	0.67	0.17
69.448	C ₄ - substituted dibenzothiophene	0.61	0.16
70.038	C ₁₂ - substituted benzene	0.69	0.18
70.199	C ₅ - substituted dibenzothiophene	0.42	0.11
70.907	C ₆ - substituted phenanthrene	0.79	0.20
71.132	C ₆ - substituted phenanthrene	0.75	0.19
71.815	C ₆ - substituted phenanthrene	0.66	0.17
71.997	C ₆ - substituted phenanthrene	0.58	0.15
73.597	C ₁₃ - substituted benzene	0.46	0.12
74.990	C ₇ - substituted phenanthrene	0.64	0.16
75.657	C ₇ - substituted phenanthrene	0.72	0.19
75.833	C ₇ - substituted phenanthrene	0.67	0.17
80.210	C ₁₅ - substituted benzene	0.24	0.06

NONPOLAR FRACTION OF FUEL NO. 16058

Analysis Summary

normal paraffins	-	24.62%	
branched paraffins	-	21.88%	
cyclo paraffins	-	<u>4.32%</u>	TOTAL 50.82%
c-C ₆ + C ₈	-	0.95%	
c-C ₆ + C ₉	-	1.40%	
c-C ₆ + C ₁₁	-	0.65%	
c-C ₆ + C ₁₂	-	0.73%	
c-C ₆ + C ₁₃	-	0.26%	
c-C ₆ + C ₁₄	-	0.33%	

NONPOLAR FRACTION OF FUEL NO. 16058 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
29.776	n-C ₁₀	0.12	0.09
35.525	n-C ₁₁	0.40	0.30
40.960	n-C ₁₂	1.30	0.97
41.615	i-C ₁₃	0.45	0.33
44.103	i-C ₁₃	0.50	0.37
44.588	i-C ₁₃	0.74	0.55
46.052	n-C ₁₃	2.57	1.91
46.758	i-C ₁₃	0.38	0.28
48.021	c-C ₆ + C ₈	0.44	0.33
48.687	i-C ₁₄	0.42	0.31
48.930	i-C ₁₄	0.69	0.51
49.240	i-C ₁₄	0.46	0.34
49.576	i-C ₁₅	1.12	0.83
50.804	n-C ₁₄	3.76	2.79
52.031	c-C ₆ + C ₉	0.82	0.61
52.834	c-C ₆ + C ₈	0.83	0.62
53.212	i-C ₁₅	0.33	0.25
53.483	i-C ₁₅	1.77	1.32
53.666	i-C ₁₅	0.28	0.21
53.780	i-C ₁₅	0.55	0.41
55.252	n-C ₁₅	4.05	3.01
57.017	i-C ₁₅	0.36	0.27
57.110	i-C ₁₄	0.33	0.24
57.336	c-C ₆ + C ₉	1.06	0.79
57.496	i-C ₁₅	0.32	0.24
57.740	i-C ₁₅	0.62	0.46
57.833	i-C ₁₆	0.46	0.34
58.034	i-C ₁₆	0.47	0.35
58.882	i-C ₁₆	0.69	0.51
59.447	n-C ₁₆	4.05	3.01
59.560	i-C ₁₇	0.29	0.22
61.322	i-C ₁₇	2.55	1.89
61.592	c-C ₆ + C ₁₁	0.88	0.65
61.795	i-C ₁₇	0.71	0.53
62.085	i-C ₁₇	0.73	0.54
63.411	n-C ₁₇	3.96	2.94
63.639	i-C ₁₈	2.61	1.94
64.916	i-C ₁₈	1.14	0.85
65.236	i-C ₁₈	0.73	0.54
65.416	i-C ₁₈	0.37	0.27
65.618	c-C ₆ + C ₁₂	0.98	0.73
65.911	i-C ₁₈	0.57	0.42
67.148	n-C ₁₈	3.58	2.66
67.487	i-C ₁₉	2.14	1.59
68.250	i-C ₁₉	0.54	0.40
68.495	i-C ₁₉	0.66	0.49
69.042	i-C ₁₉	0.47	0.35

NONPOLAR FRACTION OF FUEL NO. 16058 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
69.243	i-C ₁₉	0.58	0.43
69.422	c-C ₆ + C ₁₃	0.35	0.26
69.535	i-C ₁₉	0.44	0.33
70.296	i-C ₁₉	1.07	0.80
70.703	n-C ₁₉	3.20	2.38
71.045	i-C ₂₀	0.47	0.35
71.943	i-C ₂₀	0.64	0.48
72.503	i-C ₂₀	0.37	0.27
72.706	i-C ₂₀	0.38	0.28
72.978	c-C ₆ + C ₁₄	0.45	0.33
74.078	n-C ₂₀	2.73	2.03
75.978	i-C ₂₁	0.31	0.23
76.247	i-C ₂₁	0.28	0.21
76.407	i-C ₂₁	0.47	0.35
77.252	n-C ₂₁	1.51	1.12
80.299	n-C ₂₂	0.76	0.56
83.249	n-C ₂₃	0.41	0.30
86.097	n-C ₂₄	0.28	0.21
88.817	n-C ₂₅	0.20	0.15
91.467	n-C ₂₆	0.15	0.11
94.011	n-C ₂₇	0.11	0.08

POLAR FRACTION OF FUEL NO. 15978

Analysis Summary

C ₃ - substituted benzenes	-	0.15%		
C ₄ - substituted benzenes	-	0.20%		
C ₅ - substituted benzenes	-	0.57%		
C ₆ - substituted benzenes	-	0.18%		
C ₇ - substituted benzenes	-	0.13%		
c-C ₇ - substituted benzenes	-	0.13%		
C ₈ - substituted benzenes	-	0.23%		
C ₉ - substituted benzenes	-	0.34%		
C ₁₁ - substituted benzenes	-	0.45%		
C ₁₂ - substituted benzenes	-	0.90%		
C ₁₃ - substituted benzenes	-	0.27%		
C ₁₄ - substituted benzenes	-	0.07%		
C ₁₅ - substituted benzenes	-	0.08%	Subtotal	3.70%
tetralin		0.11%		
C ₂ - substituted tetralins	-	0.36%		
C ₃ - substituted tetralins	-	0.90%		
C ₄ - substituted tetralins	-	0.28%	Subtotal	1.65%
azulene	-	0.13%	Subtotal	0.13%
C ₄ - substituted naphthalenes	-	0.80%		
C ₅ - substituted naphthalenes	-	2.50%		
C ₆ - substituted naphthalenes	-	5.20%		
C ₇ - substituted naphthalenes	-	1.14%	Subtotal	9.64%
C ₃ - substituted dibenzofurans	-	0.13%		
C ₄ - substituted dibenzofurans	-	0.99%	Subtotal	1.12%
C ₃ - substituted fluorenes	-	0.12%	Subtotal	0.12%
C ₆ - substituted biphenyls	-	0.28%	Subtotal	0.28%
C ₄ - substituted dibenzothiophenes	-	0.78%	Subtotal	0.78%
C ₅ - substituted phenanthrenes	-	0.35%		
C ₆ - substituted phenanthrenes	-	0.15%		
C ₇ - substituted phenanthrenes	-	0.14%	Subtotal	0.64%
C ₆ - substituted anthracenes	-	0.21%	Subtotal	0.21%
			TOTAL	18.27%

POLAR FRACTION OF FUEL NO. 15978 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
27.581	1-ethyl-2-methylbenzene	0.05	0.02
29.522	1,2,3-trimethylbenzene	0.12	0.04
31.188	1-ethyl-2,3-dimethylbenzene	0.09	0.03
32.922	1-methyl-3-propylbenzene	0.17	0.06
33.194	butylbenzene	0.16	0.05
34.503	ethyl-dimethylbenzene	0.18	0.06
34.900	C ₅ - substituted benzene	0.18	0.06
35.953	C ₅ - substituted benzene	0.21	0.07
36.637	C ₅ - substituted benzene	0.15	0.05
36.887	C ₅ - substituted benzene	0.19	0.06
37.803	C ₅ - substituted benzene	0.18	0.06
38.202	C ₅ - substituted benzene	0.22	0.07
38.607	C ₅ - substituted benzene	0.40	0.13
38.856	C ₅ - substituted benzene	0.20	0.07
39.125	tetralin	0.33	0.11
40.320	azulene	0.40	0.13
40.546	dimethyl tetralin	0.15	0.05
40.777	C ₆ - substituted benzene	0.23	0.08
41.094	C ₆ - substituted benzene	0.31	0.10
42.036	C ₂ - substituted tetralin	0.16	0.05
42.437	C ₂ - substituted tetralin	0.41	0.14
43.004	C ₂ - substituted tetralin	0.35	0.12
43.728	C ₃ - substituted tetralin	0.39	0.13
44.201	C ₇ - substituted benzene	0.39	0.13
44.592	C ₃ - substituted tetralin	0.67	0.33
46.278	C ₄ - substituted naphthalene	1.90	0.42
47.087	C ₄ - substituted naphthalene	1.16	0.38
47.341	C ₃ - substituted tetralin	0.99	0.33
48.300	c-C ₇ - substituted benzene	0.40	0.13
48.536	C ₃ - substituted tetralin	0.33	0.11
49.175	C ₄ - substituted tetralin	0.84	0.28
50.189	C ₅ - substituted naphthalene	1.36	0.45
50.987	C ₅ - substituted naphthalene	1.12	0.37
51.055	C ₅ - substituted naphthalene	0.55	0.18
51.643	C ₅ - substituted naphthalene	1.52	0.50
52.446	C ₅ - substituted naphthalene	2.53	0.83
52.564	C ₅ - substituted naphthalene	0.52	0.17
52.707	C ₈ - substituted benzene	0.71	0.23
53.224	C ₆ - substituted naphthalene	1.24	0.41
53.342	C ₆ - substituted naphthalene	0.24	0.08
53.504	C ₈ - substituted benzene	0.56	0.18
53.880	C ₆ - substituted naphthalene	0.86	0.28
55.128	C ₆ - substituted naphthalene	1.37	0.45
55.554	C ₉ - substituted benzene	1.02	0.34
56.058	C ₆ - substituted naphthalene	1.52	0.50
56.222	C ₆ - substituted naphthalene	0.39	0.13
56.392	C ₆ - substituted naphthalene	0.31	0.10

POLAR FRACTION OF FUEL NO. 15978 (Cont'd)

RT	ID	Amt.	Corr. Amt.
56.610	C ₃ - substituted dibenzofuran	0.38	0.13
56.826	C ₆ - substituted naphthalene	0.81	0.27
57.018	C ₆ - substituted naphthalene	0.80	0.26
57.280	C ₆ - substituted naphthalene	0.94	0.31
57.495	C ₆ - substituted naphthalene	0.59	0.19
57.901	C ₆ - substituted naphthalene	1.00	0.33
58.043	C ₆ - substituted naphthalene	0.74	0.24
58.547	C ₆ - substituted naphthalene	1.35	0.45
58.693	C ₆ - substituted naphthalene	0.41	0.14
59.293	C ₆ - substituted naphthalene	0.50	0.17
59.340	C ₆ - substituted naphthalene	0.19	0.06
59.437	C ₃ or C ₄ - substituted fluorene	0.37	0.12
59.603	C ₆ - substituted naphthalene	0.52	0.17
59.988	C ₆ - substituted naphthalene	1.60	0.53
60.242	C ₆ - substituted naphthalene	0.39	0.13
60.338	C ₇ - substituted naphthalene	0.36	0.12
60.433	C ₁₁ - substituted benzene	0.67	0.22
60.947	C ₇ - substituted naphthalene	0.90	0.30
61.526	C ₄ - substituted dibenzofuran	1.16	0.38
62.035	C ₇ - substituted naphthalene	1.21	0.40
62.669	C ₁₁ - substituted benzene	0.70	0.23
62.863	C ₄ - substituted dibenzofuran	0.66	0.22
63.541	C ₇ - substituted naphthalene	0.51	0.17
63.587	C ₇ - substituted naphthalene	0.45	0.15
64.435	C ₄ - substituted dibenzofuran	1.17	0.39
64.660	C ₆ - substituted biphenyl	0.53	0.17
65.174	C ₆ - substituted biphenyl	0.32	0.11
65.440	C ₁₂ - substituted benzene	0.91	0.30
66.097	C ₄ - substituted dibenzothiophene	0.60	0.20
66.173	C ₄ - substituted dibenzothiophene	0.21	0.07
66.246	C ₄ - substituted dibenzothiophene	0.52	0.17
66.660	C ₁₂ - substituted benzene	0.39	0.13
67.303	C ₅ - substituted phenanthrene	1.06	0.35
69.339	C ₁₂ - substituted benzene	0.58	0.19
69.728	C ₁₂ - substituted benzene	0.44	0.15
69.903	C ₄ - substituted dibenzothiophene	0.55	0.18
70.395	C ₁₂ - substituted benzene	0.38	0.13
70.540	C ₄ - substituted dibenzothiophene	0.47	0.16
71.334	C ₆ - substituted anthracene	0.65	0.21
71.554	C ₆ - substituted phenanthrene	0.46	0.15
73.305	C ₁₃ - substituted benzene	0.37	0.12
73.997	C ₁₃ - substituted benzene	0.46	0.15
74.067	C ₁₄ - substituted benzene	0.20	0.07
75.323	C ₇ - substituted phenanthrene	0.16	0.05
75.959	C ₇ - substituted phenanthrene	0.28	0.09
80.516	C ₁₅ - substituted benzene	0.23	0.08

NONPOLAR FRACTION FROM FUEL NO. 15978

Analysis Summary

normal paraffins	-	20.71%	
branched paraffins	-	25.32%	
cyclo paraffins	-	2.23%	
olefins-		<u>0.20%</u>	TOTAL 48.56%
c-C ₅ + C ₇	-	0.19%	
c-C ₅ + C ₈	-	0.22%	
c-C ₆ + C ₄	-	0.09%	
c-C ₆ + C ₅	-	0.24%	
c-C ₆ + C ₇	-	0.34%	
c-C ₆ + C ₈	-	0.20%	
c-C ₆ + C ₉	-	0.68%	
c-C ₆ + C ₁₀	-	0.22%	
c-C ₆ + C ₁₃	-	0.15%	
6-tridecene	-	0.20%	

NONPOLAR FRACTION OF FUEL NO. 15978 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
16.032	n-C ₉	0.1	0.07
19.708	i-C ₁₀	0.05	0.03
21.821	n-C ₁₀	0.47	0.31
23.068	i-C ₁₁	0.20	0.13
23.550	c-C ₆ + C ₄	0.09	0.06
23.946	i-C ₁₁	0.13	0.09
24.984	i-C ₁₁	0.10	0.07
25.158	i-C ₁₁	0.09	0.06
25.378	i-C ₁₁	0.18	0.12
25.729	i-C ₁₁	0.14	0.09
27.500	n-C ₁₁	1.24	0.83
28.302	--	0.19	0.13
28.833	--	0.31	0.21
29.254	c-C ₆ + C ₅	0.24	0.16
30.341	i-C ₁₂	0.36	0.24
30.561	i-C ₁₂	0.22	0.15
30.807	i-C ₁₂	0.41	0.27
31.145	i-C ₁₂	0.30	0.20
31.999	c-C ₅ + C ₇	0.29	0.19
32.874	n-C ₁₂	1.97	1.32
33.480	i-C ₁₃	0.71	0.48
33.980	c-C ₆ + C ₇	0.25	0.17
34.723	c-C ₆ + C ₇	0.25	0.17
34.859	6-tridecene	0.30	0.20
35.420	i-C ₁₃	0.58	0.39
35.674	i-C ₁₃	0.33	0.22
35.947	i-C ₁₃	0.67	0.49
36.424	i-C ₁₄	0.79	0.53
37.233	c-C ₅ + C ₈	0.33	0.22
37.919	n-C ₁₃	2.83	1.90
38.282	i-C ₁₄	0.39	0.26
38.601	i-C ₁₄	0.51	0.34
39.852	c-C ₆ + C ₈	0.30	0.20
39.920	i-C ₁₄	0.28	0.19
40.104	i-C ₁₄	0.32	0.21
40.261	i-C ₁₄	0.36	0.24
40.490	i-C ₁₄	0.47	0.31
40.763	i-C ₁₄	0.78	0.52
41.083	i-C ₁₄	0.62	0.42
41.431	i-C ₁₅	1.11	0.74
42.661	n-C ₁₄	3.57	2.39
42.888	i-C ₁₅	0.24	0.16
43.163	i-C ₁₅	0.29	0.19
43.899	i-C ₁₅	0.43	0.29
44.652	c-C ₆ + C ₉	1.02	0.68
44.790	i-C ₁₅	0.36	0.24

NONPOLAR FRACTION OF FUEL NO. 15978 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
45.042	i-C ₁₅	0.39	0.26
45.322	i-C ₁₅	1.76	1.18
45.619	i-C ₁₅	0.62	0.42
46.009	i-C ₁₆	0.69	0.46
46.650	i-C ₁₅	0.27	0.18
47.108	n-C ₁₅	3.58	2.40
47.275	i-C ₁₆	0.39	0.26
48.853	i-C ₁₆	0.45	0.30
48.946	i-C ₁₆	0.31	0.21
49.184	i-C ₁₆	0.96	0.64
49.319	c-C ₆ + C ₁₀	0.33	0.22
49.571	i-C ₁₆	0.70	0.47
49.665	i-C ₁₆	0.41	0.27
49.971	i-C ₁₆	0.47	0.32
50.693	i-C ₁₆	0.61	0.41
51.301	n-C ₁₆	3.52	2.36
51.413	i-C ₁₇	0.34	0.16
51.528	i-C ₁₇	0.35	0.23
53.142	i-C ₁₇	2.16	1.45
53.392	i-C ₁₇	0.75	0.50
53.624	i-C ₁₇	0.69	0.46
53.900	i-C ₁₇	0.63	0.42
55.283	n-C ₁₇	3.41	2.28
55.469	i-C ₁₈	1.58	1.06
56.730	i-C ₁₈	1.03	0.69
57.059	i-C ₁₈	0.62	0.42
57.221	i-C ₁₈	0.35	0.23
57.427	i-C ₁₈	0.94	0.63
57.725	i-C ₁₈	0.48	0.32
58.996	n-C ₁₈	3.13	2.10
59.324	i-C ₁₉	1.89	1.27
60.069	i-C ₁₉	0.53	0.36
60.300	i-C ₁₉	0.59	0.40
60.462	i-C ₁₉	0.27	0.18
60.623	i-C ₁₉	0.33	0.22
61.065	i-C ₁₉	0.53	0.36
61.227	c-C ₆ + C ₁₃	0.23	0.15
61.342	i-C ₁₉	0.38	0.25
62.097	i-C ₁₉	0.75	0.50
62.514	n-C ₁₉	2.43	1.63
62.860	i-C ₂₀	0.39	0.26
63.737	i-C ₂₀	0.50	0.34
63.919	i-C ₂₀	0.23	0.15
64.309	i-C ₂₀	0.28	0.19
64.492	i-C ₂₁	0.28	0.19
64.628	i-C ₂₀	0.21	0.14
64.790	i-C ₂₀	0.33	0.22

NONPOLAR FRACTION OF FUEL NO. 15978 (Cont'd)

<u>Retention Time</u>	<u>Peak Identification</u>	<u>Fraction Amount</u>	<u>Sample Amount</u>
65.871	n-C ₂₀	1.99	1.33
66.997	i-C ₂₁	0.26	0.17
67.591	i-C ₂₁	0.21	0.14
67.773	i-C ₂₁	0.25	0.15
68.046	i-C ₂₁	0.22	0.15
68.205	i-C ₂₁	0.34	0.23
69.055	n-C ₂₁	1.18	0.79
72.108	n-C ₂₂	0.77	0.52
75.053	n-C ₂₃	0.41	0.27
77.875	n-C ₂₄	0.21	0.14
80.599	n-C ₂₅	0.10	0.07

POLAR FRACTION OF FUEL NO. 16082

Analysis Summary

C ₄ - substituted benzenes	-	3.33%		
C ₅ - substituted benzenes	-	2.46%		
C ₆ - substituted benzenes	-	1.20%		
C ₇ - substituted benzenes	-	0.25%		
C ₁₀ - substituted benzenes	-	0.15%		
C ₁₁ - substituted benzenes	-	0.11%		
C ₁₂ - substituted benzenes	-	0.23%		
C ₁₃ - substituted benzenes	-	0.21%		
C ₁₄ - substituted benzenes	-	0.45%		
C ₁₈ - substituted benzenes	-	0.24%	Subtotal	8.63%
substituted indene + dihydroindenes	-	0.63%	Subtotal	0.63%
tetralin	-	0.63%		
C ₂ - substituted tetralins	-	0.77%		
C ₃ - substituted tetralins	-	0.10%		
C ₄ - substituted tetralins	-	0.41%		
C ₅ - substituted tetralins	-	0.20%		
C ₆ - substituted tetralins	-	0.46%		
C ₇ - substituted tetralins	-	0.18%	Subtotal	2.75%
C ₃ - substituted naphthalenes	-	2.65%		
C ₄ - substituted naphthalenes	-	2.13%		
C ₅ - substituted naphthalenes	-	0.33%	Subtotal	5.11%
biphenyl + substituted biphenyls	-	0.30%	Subtotal	0.30%
C ₆ - substituted fluorenes	-	0.23%	Subtotal	0.23%
C ₅ - substituted dibenzothiophenes	-	0.07%	Subtotal	0.07%
			TOTAL	17.72%

POLAR FRACTION OF FUEL NO. 16082 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
30.962	diethylbenzene	0.20	0.09
32.727	C ₄ - substituted benzene	0.53	0.24
32.923	C ₄ - substituted benzene	0.28	0.12
32.991	C ₄ - substituted butylbenzene	0.20	0.09
33.209	C ₄ - substituted 4-ethyl-1,2-dimethylbenzene	0.79	0.35
33.598	C ₅ - substituted benzene	0.35	0.16
34.266	C ₄ - substituted benzene	0.67	0.30
34.376	C ₄ - substituted benzene	0.73	0.32
34.744	C ₅ - substituted benzene	1.26	0.56
35.031	C ₅ - substituted benzene	0.27	0.12
35.569	C ₅ - substituted benzene	0.40	0.18
35.744	C ₅ - substituted benzene	0.52	0.23
35.876	C ₅ - substituted benzene	0.44	0.20
36.506	C ₄ - substituted benzene	1.10	0.49
36.753	C ₄ - substituted benzene	2.98	1.33
37.466	C ₅ - substituted benzene	0.13	0.06
37.532	C ₅ - substituted benzene	0.15	0.07
37.619	C ₂ - substituted indene	0.34	0.15
37.944	C ₅ - substituted benzene	0.51	0.23
38.055	C ₅ - substituted benzene	0.40	0.18
38.273	substituted 2,3-dihydro-1-methylindene	0.46	0.20
38.447	C ₆ - substituted benzene	0.94	0.42
38.692	C ₅ - substituted benzene	0.25	0.11
38.867	C ₅ - substituted benzene	0.36	0.16
38.997	tetralin	1.42	0.63
39.716	C ₅ - substituted benzene	0.46	0.20
40.174	substituted naphthalene	1.43	0.64
40.306	substituted azulene	1.63	0.73
40.400	tri substituted dihydroindene	0.33	0.15
40.531	C ₆ - substituted benzene	0.45	0.20
40.793	C ₆ - substituted benzene	0.53	0.24
40.904	C ₆ - substituted benzene	0.17	0.08
41.080	C ₃ - substituted dihydroindene	0.29	0.13
41.406	C ₆ - substituted benzene	0.19	0.08
41.604	dimethyl tetralin	0.32	0.14
41.757	dimethyl tetralin	0.34	0.15
42.677	C ₆ - substituted benzene	0.24	0.11
43.420	C ₃ - tetralin	0.31	0.14
43.899	C ₆ - substituted benzene	0.16	0.07
44.320	dimethyl tetralin	1.07	0.48
46.037	C ₃ - substituted naphthalene	4.26	1.90
46.763	C ₃ - substituted naphthalene	1.69	0.75
46.960	C ₇ - substituted benzene	0.57	0.25
47.841	C ₃ - substituted tetralin	0.18	0.08
49.737	biphenyl	0.31	0.14
50.442	C ₄ - substituted naphthalene	0.43	0.19

POLAR FRACTION OF FUEL NO. 16082 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
51.040	C ₄ - substituted naphthalene	1.24	0.55
51.707	C ₄ - substituted naphthalene	0.98	0.44
51.885	C ₄ - substituted naphthalene	0.64	0.28
52.550	C ₄ - substituted naphthalene	0.31	0.14
52.683	C ₄ - substituted naphthalene	0.28	0.12
53.213	C ₄ - substituted naphthalene	0.24	0.26
54.504	C ₂ ? - substituted biphenyl	0.35	0.16
55.376	C ₄ - substituted naphthalene	0.34	0.15
56.122	C ₄ - substituted tetralin	0.48	0.21
56.332	C ₄ - substituted tetralin	0.46	0.20
56.548	C ₅ - substituted naphthalene	0.44	0.20
56.861	C ₅ - substituted naphthalene	0.30	0.13
57.221	C ₆ - substituted tetralin	0.30	0.13
57.358	C ₆ - substituted tetralin	0.27	0.12
57.945	C ₆ - substituted tetralin	0.48	0.21
58.011	C ₁₀ - substituted benzene	0.33	0.15
58.983	C ₇ - substituted tetralin	0.41	0.18
62.205	C ₁₁ - substituted benzene	0.25	0.11
63.737	C ₆ - substituted fluorene	0.51	0.23
64.836	--	0.75	0.33
65.261	C ₅ - substituted dibenzothiophene	0.15	0.07
66.196	C ₁₂ - substituted benzene	0.51	0.23
66.633	C ₁₃ - substituted benzene	0.48	0.21
69.341	C ₁₄ - substituted benzene	0.51	0.23
69.988	C ₁₄ - substituted benzene	0.50	0.22
80.231	C ₁₈ - substituted benzene	0.53	0.24

NONPOLAR FRACTION OF FUEL NO. 16082

Analysis Summary

normal paraffins	-	14.71%	
branched paraffins	-	25.72%	
cyclo paraffins	-	0.28%	
olefins	-	<u>1.18%</u>	TOTAL 41.89%
c-C ₆ + C ₇	-	0.04%	
c-C ₆ + C ₈	-	0.24%	
C ₁₁ - olefin	-	0.03%	
C ₁₄ - olefin	-	0.06%	
C ₁₅ - olefin	-	0.07%	
C ₁₆ - olefin	-	0.56%	
C ₁₆ - olefin	-	0.46%	

NONPOLAR FRACTION OF FUEL NO. 16082 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
33.359	i-C ₁₁	0.05	0.03
35.436	n-C ₁₁	0.47	0.26
36.844	C ₁₁ - olefin	0.06	0.03
38.819	i-C ₁₂	0.08	0.04
39.156	i-C ₁₂	0.05	0.03
40.773	C ₁₂	0.53	0.29
41.425	i-C ₁₃	0.16	0.09
43.929	i-C ₁₃	0.10	0.06
44.378	i-C ₁₃	0.16	0.09
45.773	n-C ₁₃	0.63	0.35
46.582	i-C ₁₄	0.12	0.07
47.824	c-C ₆ + C ₇	0.08	0.04
48.480	i-C ₁₄	0.13	0.07
48.737	i-C ₁₄	0.14	0.08
49.054	i-C ₁₄	0.14	0.08
49.371	i-C ₁₄	0.38	0.21
50.517	n-C ₁₄	1.13	0.63
50.632	i-C ₁₅	0.14	0.07
51.793	C ₁₅ - olefin	0.11	0.06
52.646	c-C ₆ + C ₈	0.44	0.24
52.786	i-C ₁₅	0.19	0.11
53.036	i-C ₁₅	0.21	0.12
53.269	i-C ₁₆	0.87	0.48
53.455	C ₁₅ - olefin	0.13	0.07
53.593	i-C ₁₅	0.31	0.17
53.913	i-C ₁₆	0.21	0.12
54.029	i-C ₁₅	0.20	0.11
54.658	C ₁₆ - olefin	0.14	0.56
55.030	n-C ₁₅	2.27	1.26
55.258	i-C ₁₆	0.26	0.14
56.884	i-C ₁₆	0.31	0.17
56.955	i-C ₁₆	0.21	0.12
57.971	i-C ₁₆ - olefin	0.82	0.46
57.327	i-C ₁₆	0.23	0.13
57.536	i-C ₁₆	0.48	0.27
57.678	i-C ₁₆	0.37	0.21
57.888	i-C ₁₆	0.37	0.21
58.732	i-C ₁₆	0.54	0.30
59.272	n-C ₁₆	3.01	1.67
59.413	i-C ₁₇	0.32	0.18
59.552	i-C ₁₇	0.59	0.33
60.506	i-C ₁₇	0.32	0.18
60.930	i-C ₁₇	0.39	0.22
61.165	i-C ₁₈	1.95	1.08
61.450	i-C ₁₇	0.76	0.42
61.662	i-C ₁₇	0.54	0.30
61.946	i-C ₁₇	0.67	0.37

NONPOLAR FRACTION OF FUEL NO. 16082 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
63.274	n-C ₁₇	3.47	1.93
63.511	i-C ₁₈	2.32	1.29
63.628	i-C ₁₈	0.47	0.26
64.797	i-C ₁₉	1.02	0.57
64.891	i-C ₁₈	0.24	0.13
65.082	i-C ₁₈	0.42	0.23
65.130	i-C ₁₈	0.34	0.19
65.296	i-C ₁₉	0.41	0.23
65.487	i-C ₁₉	0.86	0.48
65.797	i-C ₁₈	0.67	0.37
67.022	n-C ₁₈	3.71	2.06
67.381	i-C ₂₀	2.70	1.50
67.621	i-C ₁₉	0.32	0.18
68.172	i-C ₁₉	0.66	0.37
68.387	i-C ₂₀	0.78	0.43
68.538	i-C ₁₉	0.37	0.21
68.706	i-C ₂₀	0.52	0.29
68.947	i-C ₁₉	0.60	0.33
69.138	i-C ₁₉	0.71	0.39
69.304	i-C ₁₉	0.44	0.24
69.427	i-C ₁₉	0.61	0.34
69.739	i-C ₁₉	0.71	0.39
70.027	i-C ₂₀	0.36	0.20
70.197	i-C ₂₀	1.13	0.63
70.610	n-C ₁₉	3.20	1.78
70.732	i-C ₂₀	0.46	0.26
70.948	i-C ₂₀	0.63	0.35
71.386	i-C ₂₁	0.74	0.41
71.845	i-C ₂₁	0.87	0.48
72.011	i-C ₂₀	0.40	0.22
72.181	i-C ₂₁	0.45	0.25
72.424	i-C ₂₁	0.58	0.32
72.593	i-C ₂₁	0.54	0.30
72.746	i-C ₂₀	0.45	0.25
72.891	i-C ₂₁	0.59	0.33
73.278	i-C ₂₁	0.92	0.51
74.007	n-C ₂₀	3.25	1.80
74.225	i-C ₂₁	0.43	0.24
74.419	i-C ₂₂	0.52	0.29
74.516	i-C ₂₂	0.47	0.26
74.934	i-C ₂₂	0.45	0.25
75.130	i-C ₂₂	0.65	0.36
75.249	i-C ₂₂	0.26	0.14
75.492	i-C ₂₂	0.50	0.28
75.713	i-C ₂₂	0.58	0.32
75.908	i-C ₂₂	0.62	0.34
76.173	i-C ₂₂	0.59	0.33

NONPOLAR FRACTION OF FUEL NO. 16082 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
76.343	i-C ₂₃	0.93	0.52
77.199	n-C ₂₁	2.44	1.35
77.515	i-C ₂₂	0.42	0.23
78.097	i-C ₂₃	0.69	0.38
78.293	i-C ₂₃	0.37	0.21
78.347	i-C ₂₂	0.37	0.21
78.658	i-C ₂₃	0.39	0.22
78.879	i-C ₂₃	0.44	0.24
79.053	i-C ₂₃	0.52	0.29
79.319	i-C ₂₃	0.60	0.33
79.588	i-C ₂₃	0.68	0.38
80.254	n-C ₂₂	1.65	0.92
81.278	i-C ₂₄	0.70	0.39
82.044	C ₂₂ ?	0.22	0.12
83.162	n-C ₂₃	0.73	0.41
85.954	i-C ₂₅	0.17	0.09

POLAR FRACTION OF FUEL NO. 15980

Analysis Summary

C ₁ - dihydroindene	-	0.07%		
C ₂ - dihydroindene	-	0.36%	Subtotal	0.43%
naphthalene	-	0.17%		
C ₂ - substituted naphthalenes	-	8.49%		
C ₄ - substituted naphthalenes	-	3.00%		
C ₅ - substituted naphthalenes	-	5.57%		
C ₆ - substituted naphthalenes	-	0.97%	Subtotal	18.20%
C ₂ - substituted tetralins	-	0.30%	Subtotal	0.30%
C ₇ - substituted benzenes	-	0.76%		
C ₁₁ - substituted benzenes	-	0.48%		
C ₁₂ - substituted benzenes	-	0.73%	Subtotal	1.97%
C ₄ - substituted biphenyls	-	0.26%		
C ₅ - substituted biphenyls	-	2.89%		
C ₆ - substituted biphenyls	-	1.19%	Subtotal	4.34%
C ₃ - substituted azulenes	-	0.40%	Subtotal	0.40%
C ₂ - substituted fluorenes	-	1.59%		
C ₃ - substituted fluorenes	-	0.19%	Subtotal	1.78%
C ₃ - substituted dibenzothiophenes	-	1.09%	Subtotal	1.09%
C ₂ - substituted phenanthrenes	-	3.67%		
C ₃ - substituted phenanthrenes	-	0.73%		
C ₄ - substituted phenanthrenes	-	1.16%		
C ₅ - substituted phenanthrenes	-	2.30%		
C ₆ - substituted phenanthrenes	-	0.20%		
C ₇ - substituted phenanthrenes	-	0.75%		
C ₈ - substituted phenanthrenes	-	0.19%	Subtotal	9.00%
C ₃ - substituted anthracenes	-	1.49%		
C ₄ - substituted anthracenes	-	0.42%		
C ₅ - substituted anthracenes	-	0.22%	Subtotal	2.13%
C ₂ - substituted fluoranthenes	-	0.49%		
C ₃ - substituted fluoranthenes	-	0.60%	Subtotal	1.09%
C ₃ - substituted carbazole	-	0.30%	Subtotal	0.30%
C ₄ - substituted pyrene	-	0.21%	Subtotal	0.21%
C ₄ - substituted 2-phenyl naphthalene	-	0.46%	Subtotal	0.46%
C ₂ - substituted - (phenylmethyl) naphthalene	-	0.16%	Subtotal	0.16%
			TOTAL	41.86%

POLAR FRACTION OF FUEL NO. 15980 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
38.276	dihydromethylindene	0.10	0.07
40.151	naphthalene	0.26	0.17
40.285	dihydrodimethylindene	0.08	0.05
40.841	dihydrodimethylindene	0.14	0.09
43.577	dihydrodimethylindene	0.33	0.22
44.395	substituted tetralin	0.46	0.30
46.054	C ₂ - substituted naphthalene	1.34	0.88
46.169	C ₂ - substituted naphthalene	0.32	0.21
46.943	C ₂ - substituted naphthalene	1.19	0.78
47.149	C ₇ - substituted benzene	0.34	0.22
50.826	di - substituted naphthalene	1.55	1.01
51.482	di - substituted naphthalene	2.15	1.40
52.280	di - substituted naphthalene	2.46	1.61
52.396	di - substituted naphthalene	0.68	0.45
53.026	di - substituted naphthalene	1.13	0.74
53.145	di - substituted naphthalene	0.31	0.20
53.679	di - substituted naphthalene	0.75	0.49
54.895	di - substituted naphthalene	1.11	0.72
55.249	C ₇ - substituted benzene	0.88	0.54
55.866	C ₄ - substituted naphthalene	1.68	1.10
56.274	C ₄ - substituted naphthalene	0.70	0.46
56.817	C ₄ - substituted naphthalene	1.36	0.89
57.055	C ₄ - substituted naphthalene	0.84	0.55
57.832	C ₅ - substituted naphthalene	0.69	0.45
58.404	C ₅ - substituted naphthalene	1.50	0.98
59.137	C ₅ - substituted naphthalene	0.76	0.50
59.824	C ₅ - substituted naphthalene	1.57	1.03
60.084	C ₅ - substituted naphthalene	0.95	0.62
60.227	C ₅ - substituted naphthalene	0.25	0.16
60.368	C ₅ - substituted naphthalene	0.44	0.29
60.728	C ₅ - substituted naphthalene	0.71	0.46
60.893	C ₅ - substituted naphthalene	0.17	0.11
61.038	C ₅ - substituted naphthalene	0.40	0.26
61.300	C ₅ - substituted biphenyl	0.56	0.37
61.827	C ₅ - substituted naphthalene	1.09	0.71
62.190	C ₄ - substituted biphenyl	0.40	0.26
62.668	7-ethyl-1,4-dimethylazulene	0.61	0.40
63.431	C ₆ - substituted naphthalene	1.13	0.74
63.670	C ₆ - substituted naphthalene	0.35	0.23
63.839	C ₂ - substituted fluorene	0.90	0.59
64.226	C ₅ - substituted biphenyl	1.44	0.94
64.514	C ₅ - substituted biphenyl	0.79	0.52
64.710	C ₅ - substituted biphenyl	0.60	0.39
64.875	C ₅ - substituted biphenyl	0.52	0.34
65.044	C ₅ - substituted biphenyl	0.51	0.33
65.310	C ₆ - substituted biphenyl	0.97	0.63
65.502	C ₄ - substituted biphenyl	0.47	0.31

POLAR FRACTION OF FUEL NO. 15980 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
65.982	C ₆ - substituted biphenyl	0.51	0.33
66.080	C ₃ - substituted dibenzothiophene	0.31	0.20
66.136	C ₃ - substituted dibenzothiophene	0.32	0.21
66.376	C ₁₁ - substituted benzene	0.74	0.48
67.145	C ₂ - substituted fluorene	1.24	0.81
67.217	C ₂ - substituted fluorene	0.29	0.19
67.266	C ₃ - substituted fluorene	0.29	0.19
67.459	C ₂ - substituted phenanthrene	0.40	0.26
67.653	C ₂ - substituted phenanthrene	0.35	0.23
67.772	C ₂ - substituted phenanthrene	0.44	0.29
68.335	C ₂ - substituted phenanthrene	0.83	0.54
68.529	C ₂ - substituted phenanthrene	0.51	0.33
68.673	C ₂ - substituted phenanthrene	0.48	0.31
68.890	C ₂ - substituted phenanthrene	0.58	0.38
69.277	C ₁₂ - substituted benzene	0.55	0.36
69.447	C ₂ - substituted phenanthrene	0.43	0.28
69.666	C ₆ - substituted biphenyl	0.35	0.23
69.836	C ₃ - substituted dibenzothiophene	0.54	0.35
70.129	C ₁₂ - substituted benzene	0.57	0.37
70.546	C ₃ - substituted dibenzothiophene	0.50	0.33
71.395	C ₂ - substituted phenanthrene	1.61	1.05
71.685	C ₃ - substituted phenanthrene	0.58	0.38
71.905	C ₃ - substituted anthracene	0.55	0.36
72.345	C ₃ - substituted anthracene	1.27	0.83
72.544	C ₃ - substituted anthracene	0.46	0.30
72.739	C ₃ - substituted phenanthrene	0.48	0.31
72.909	C ₃ - substituted carbazole	0.46	0.30
73.326	C ₃ - substituted phenanthrene	0.64	0.42
73.867	C ₄ - substituted phenyl naphthalene	0.70	0.46
74.013	C ₄ - substituted phenanthrene	0.35	0.23
74.460	C ₄ - substituted phenanthrene	0.44	0.29
75.097	C ₄ - substituted anthracene	0.64	0.42
75.491	C ₄ - substituted phenanthrene	0.98	0.64
75.636	C ₅ - substituted phenanthrene	0.65	0.42
76.177	C ₅ - substituted phenanthrene	1.69	1.10
76.374	C ₅ - substituted phenanthrene	0.51	0.33
76.422	C ₅ - substituted phenanthrene	0.31	0.20
76.577	C ₅ - substituted phenanthrene	0.39	0.25
76.726	C ₅ - substituted phenanthrene	0.69	0.45
76.876	C ₅ - substituted anthracene	0.33	0.22
77.049	C ₂ - substituted fluoranthene	0.36	0.24
77.197	C ₆ - substituted phenanthrene	0.31	0.20
77.739	C ₂ - substituted fluoranthene	0.39	0.25
78.779	C ₃ - substituted fluoranthene	0.56	0.37

POLAR FRACTION OF FUEL NO. 15980 (Cont'd)

<u>Retention Time</u>	<u>Peak Identification</u>	<u>Fraction Amount</u>	<u>Sample Amount</u>
78.903	C ₃ - substituted fluoranthene	0.35	0.23
79.592	C ₇ - substituted phenanthrene	0.38	0.25
79.889	C ₇ - substituted phenanthrene	0.76	0.50
80.192	C ₈ - substituted phenanthrene	0.29	0.19
82.448	C ₄ - substituted pyrene	0.32	0.21
82.716	C ₂ - substituted (phenylmethyl) naphthalene	0.24	0.16

NONPOLAR FRACTION OF FUEL NO. 15980

Analysis Summary

normal paraffins	-	11.95%	
branched paraffins	-	14.16%	
cyclo-paraffins	-	<u>0.53%</u>	TOTAL 26.64%
c-C ₆ + C ₁₅	-	0.18%	
c-C ₆ + C ₁₇	-	0.35%	

NONPOLAR FRACTION OF FUEL NO. 15980 (Cont'd)

Retention Time	Peak Identification	Fraction Amount	Sample Amount
45.889	n-C ₁₃	0.26	0.09
50.663	n-C ₁₄	0.75	0.26
52.813	n-C ₁₄	0.24	0.08
53.442	i-C ₁₅	0.45	0.16
53.754	i-C ₁₅	0.21	0.09
55.156	n-C ₁₅	1.14	0.40
57.079	i-C ₁₆	0.28	0.10
57.332	i-C ₁₆	0.29	0.10
57.737	i-C ₁₆	0.37	0.13
59.050	i-C ₁₆	0.24	0.08
59.401	n-C ₁₆	1.43	0.50
61.265	i-C ₁₇	0.96	0.33
61.584	i-C ₁₇	0.33	0.11
61.811	i-C ₁₇	0.40	0.14
62.106	i-C ₁₇	0.42	0.15
63.448	n-C ₁₇	2.07	0.72
63.609	i-C ₁₉	0.98	0.34
64.961	i-C ₁₈	0.69	0.24
65.260	i-C ₁₈	0.40	0.14
65.673	i-C ₁₈	0.68	0.23
65.948	i-C ₁₈	0.48	0.17
67.241	n-C ₁₈	2.83	0.98
67.520	i-C ₂₀	1.23	0.43
68.606	i-C ₁₉	0.91	0.32
69.146	i-C ₁₉	0.45	0.16
69.333	i-C ₁₉	0.79	0.27
69.632	i-C ₁₉	0.68	0.24
70.895	n-C ₁₉	3.64	1.26
71.010	i-C ₂₀	0.38	0.13
71.200	i-C ₂₀	0.46	0.16
72.088	i-C ₂₁	0.88	0.31
72.252	i-C ₂₀	0.42	0.15
72.651	i-C ₂₀	0.57	0.20
72.868	i-C ₂₀	0.93	0.32
73.149	i-C ₂₁	0.88	0.31
74.396	n-C ₂₀	4.56	1.58
74.587	i-C ₂₁	0.26	0.09
74.728	c-C ₆ + C ₁₅	0.51	0.18
75.421	i-C ₂₂	1.20	0.42
75.635	i-C ₂₂	0.42	0.15
75.779	i-C ₂₂	0.55	0.19
76.016	i-C ₂₂	0.68	0.24
76.204	i-C ₂₂	0.99	0.34
76.470	i-C ₂₂	0.88	0.31
76.634	i-C ₂₂	1.08	0.37
76.901	--	1.08	0.37
77.685	n-C ₂₁	5.06	1.76

APPENDIX A-2
Reconstructed Ion Chromatograms
(TIC)

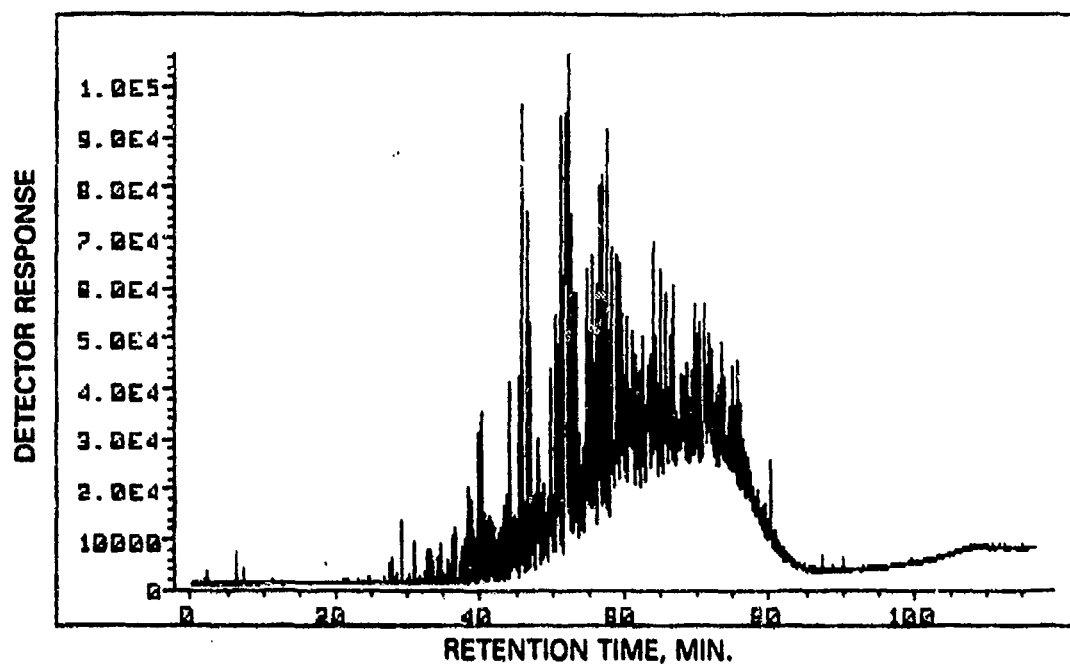


Figure A-1. Polar fraction of fuel No. 16058

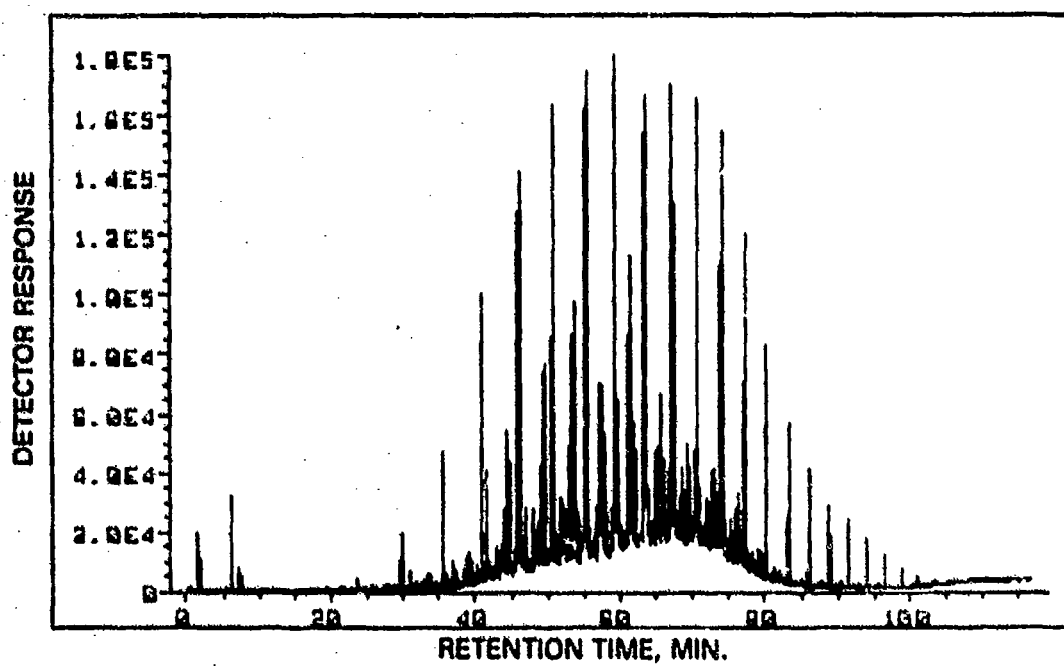


Figure A-2. Nonpolar fraction of fuel No. 16058

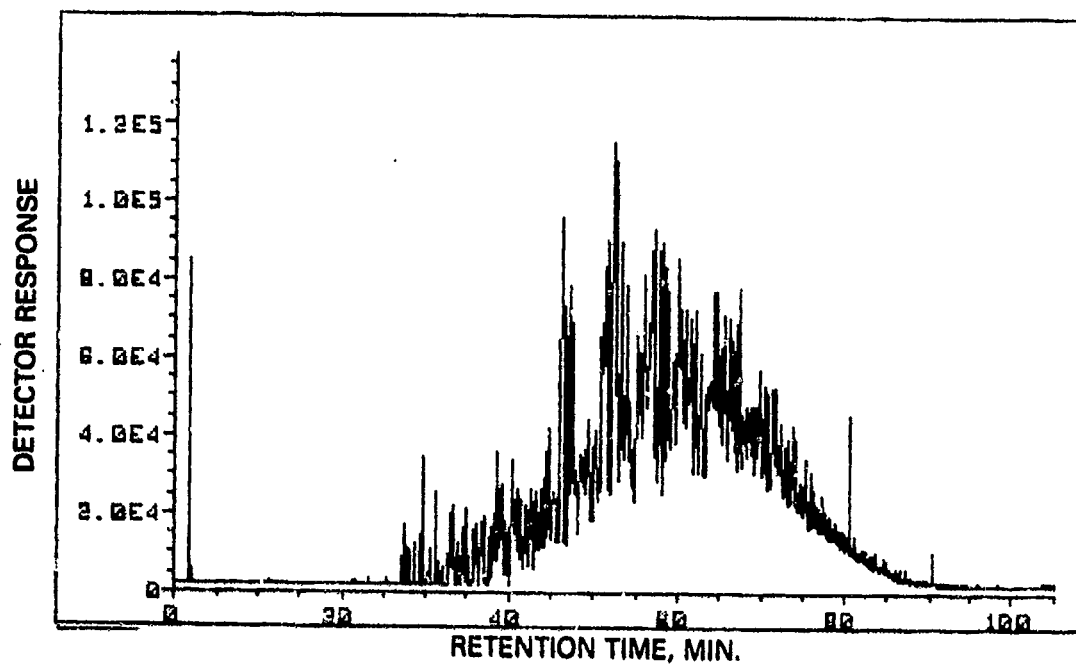


Figure A-3. Polar fraction of fuel No. 15978

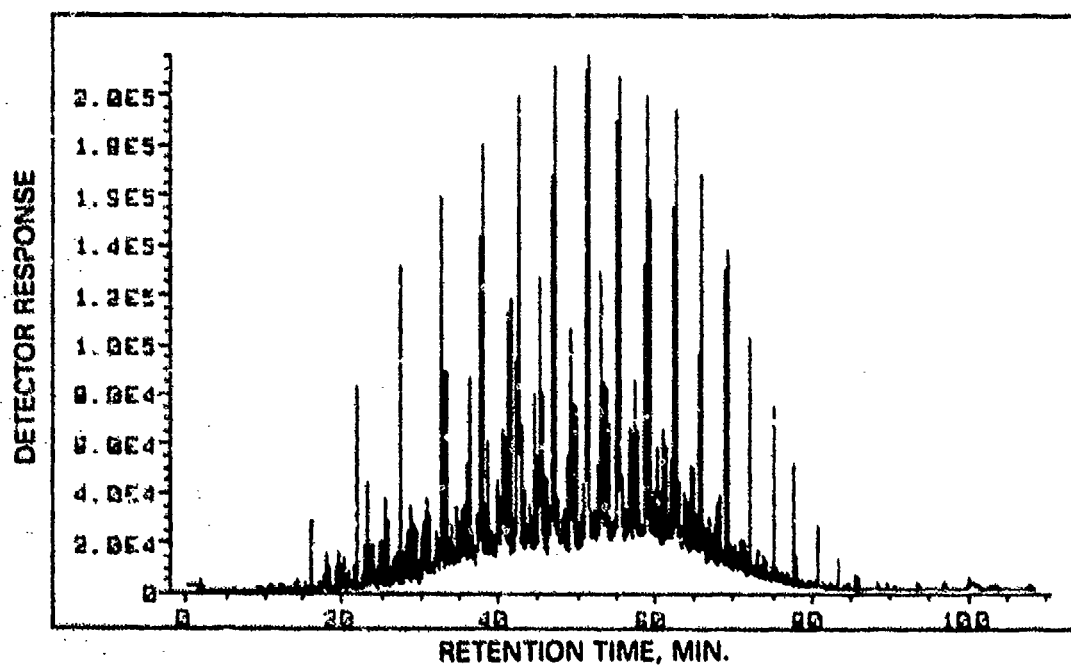


Figure A-4. Nonpolar fraction of fuel No. 15978

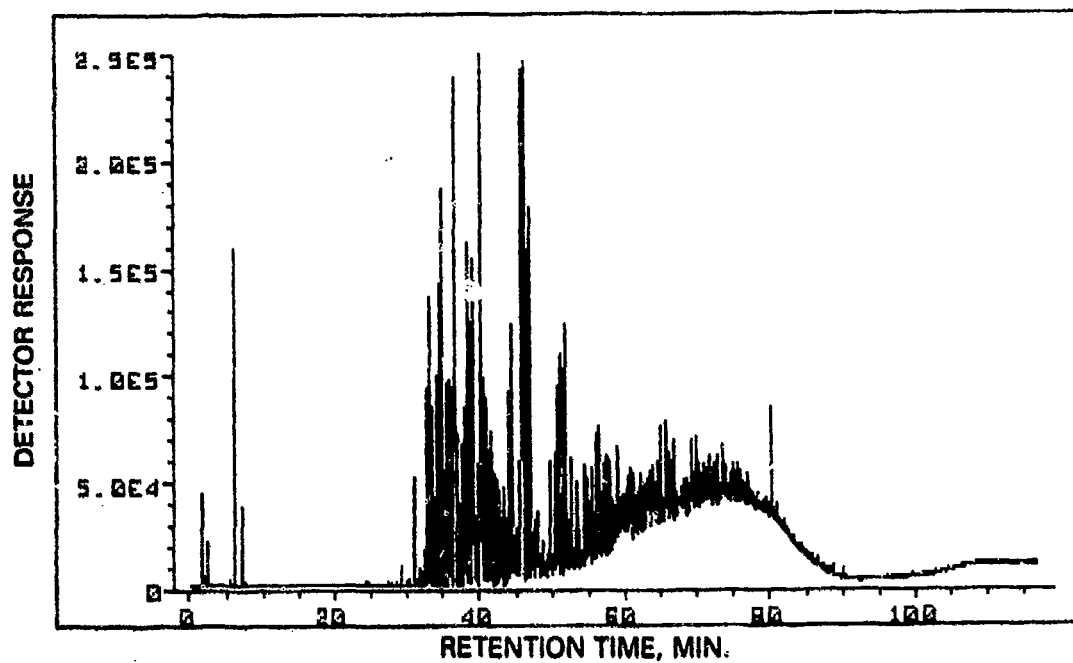


Figure A-5. Polar fraction of fuel No. 16082

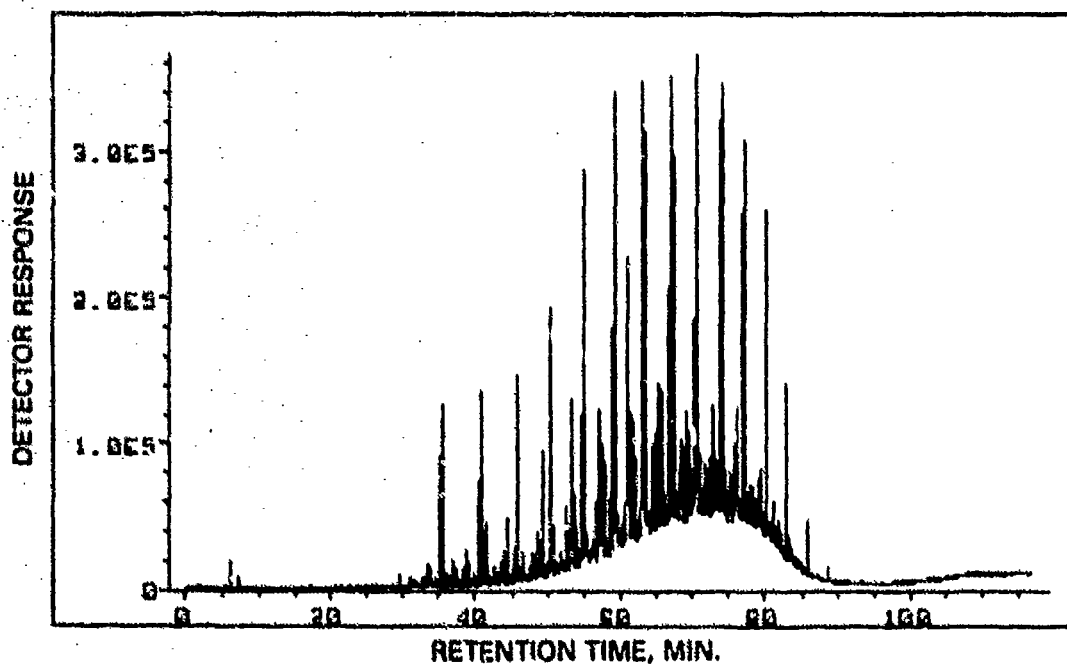


Figure A-6. Nonpolar fraction of fuel No. 16082

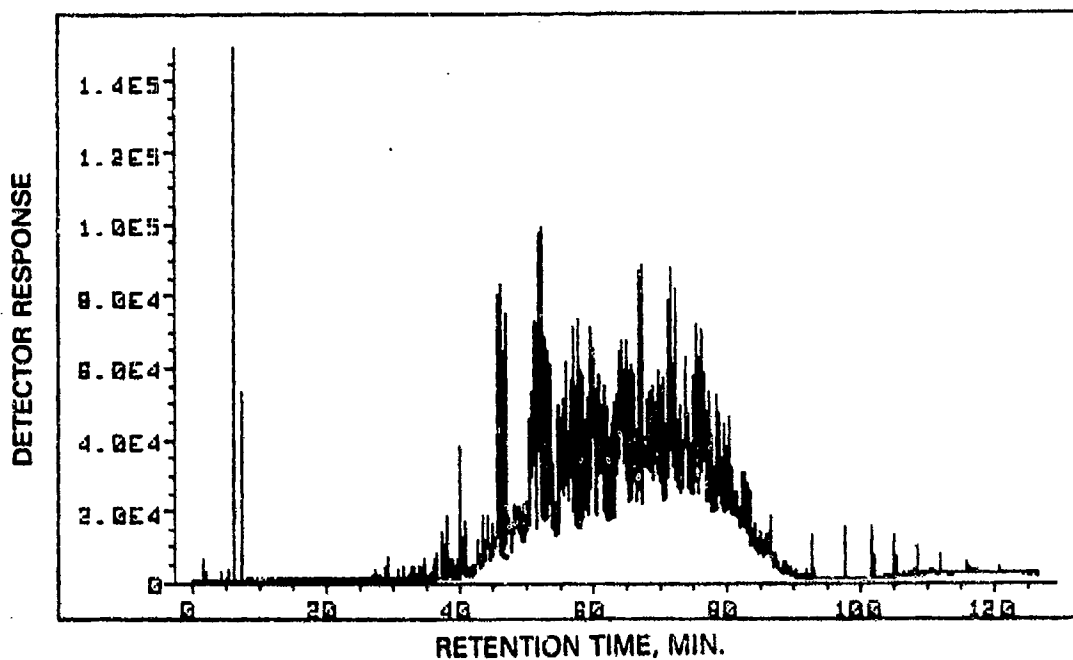


Figure A-7. Polar fraction of fuel No. 15980

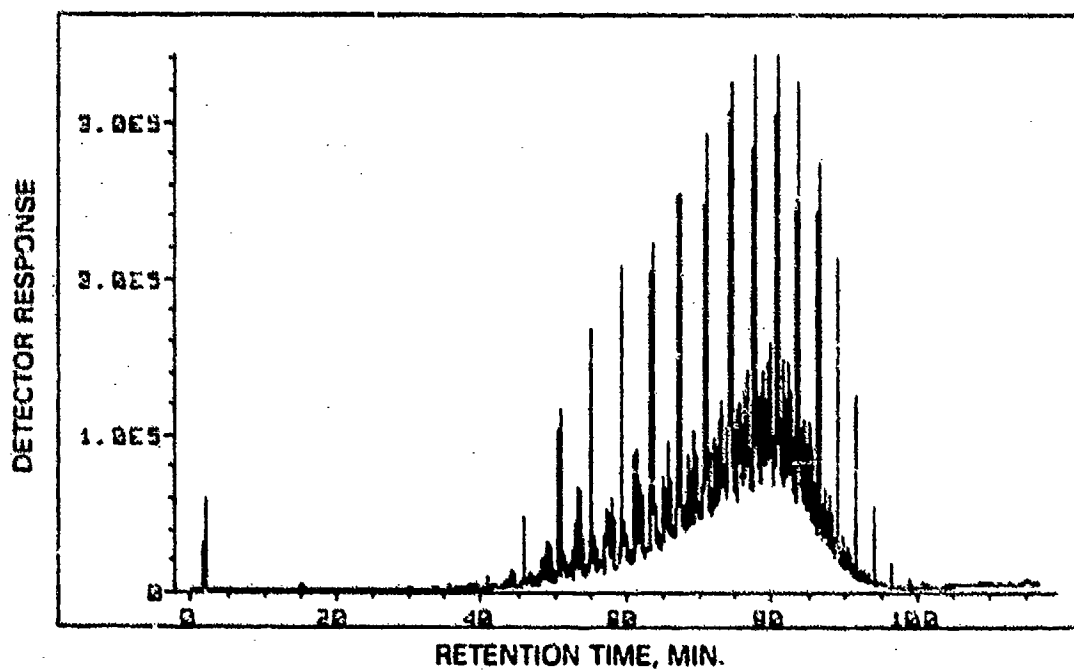


Figure A-8. Nonpolar fraction of fuel No. 15980

DISTRIBUTION LIST

DEPARTMENT OF DEFENSE

DEFENSE TECHNICAL INFORMATION
CTR
CAMERON STATION
ALEXANDRIA VA -22314

12

DEPT. OF DEFENSE
ATTN: OASD/A&L (EP)
(MR DYCKMAN)
WASHINGTON DC 20301-8000

1

DOD
ATTN: DUSDRE (RAT) (DR DIX)
ATTN: ROOM 3-D-1089, PENTAGON
WASHINGTON DC 20301

1

1

DEFENSE ADVANCED RES PROJ
AGENCY
DEFENSE SCIENCES OFC
1400 WILSON BLVD
ARLINGTON VA 22209

1

DEPARTMENT OF THE ARMY

CDR
U.S. ARMY BELVOIR RESEARCH,
DEVELOPMENT & ENGINEERING CTR
ATTN: STRBE-VF
STRBE-BT
FORT BELVOIR VA 22060-5606

10

2

HQ, DEPT OF ARMY
ATTN: DALO-TSE (COL BLISS)
DALO-TSZ-B (MR KOWALCZYK)
DALO-AV
DAMO-FDR (MAJ KNOX)
SARD-TN (DR CHURCH)
SARD-TR (MR APPEL)
WASHINGTON DC 20310-0005

1

1

1

1

1

1

CDR
US ARMY MATERIEL COMMAND
ATTN: AMCDE-SS
AMCSM-WST
5001 EISENHOWER AVE
ALEXANDRIA VA 22333-0001

1

1

CDR
US ARMY LABORATORY COMMAND
ATTN: AMSLC-TP-AL (LTC SCHRADER)
ADELPHI MD 20783-1145

1

CDR
US ARMY GENERAL MATERIAL &
PETROLEUM ACTIVITY
ATTN: STRGP-F (MR ASHBROOK)
STRGP-FE, BLDG 85-3
(MR GARY SMITH)
STRGP-FT (MR FOSTER)
NEW CUMBERLAND PA 17070-5008

1

1

1

CDR
US ARMY RES & STDZN GROUP
(EUROPE)
ATTN: AMXSN-UK-RA (DR OERTEL)
AMXSN-UK-SE
BOX 65
FPO NEW YORK 09510

1

1

CDR
US ARMY YUMA PROVING GROUND
ATTN: STEYP-MT-TL-M
(MR DOEBBLER)
YUMA AZ 85364-9103

1

CDR
CHEMICAL R&D CENTER
ATTN: SMCCR-MMS
ABERDEEN PROVING GRD MD
21010-5423

1

CDR
THEATER ARMY MATERIAL MGMT
CENTER (200TH)-DPGM
DIRECTORATE FOR PETROL MGMT
ATTN: AEAGD-MMC-PT-Q
(MR CARLONE)
APO NY 09052

1

CDR
US ARMY RESEARCH OFC
ATTN: SLCRO-EG (DR MANN)
SLCRO-CB
P O BOX 12211
RSCH TRIANGLE PARK NC 27709-2211

1

1

CDR
US ARMY DEPOT SYSTEMS CMD
ATTN: AMSDS-RM-EFO
CHAMBERSBURG PA 17201

1

HQ, EUROPEAN COMMAND
ATTN: J4/7-LJPO
VAIHINGEN, GE
APO NY 09128

1

CDR
US ARMY GENERAL MATERIAL &
PETROLEUM ACTIVITY
ATTN: STRGP-PW (MR PRICE)
BLDG 247, DEFENSE DEPOT TRACY
TRACY CA 95376-5051

CDR
US ARMY FOREIGN SCIENCE & TECH
CENTER
ATTN: AIAST-RA-ST3 (MR BUSI)
AIAST-MT-1
FEDERAL BLDG
CHARLOTTESVILLE VA 22901

HQ, US ARMY T&E COMMAND
ATTN: AMSTE-TO-O
AMSTE-CM-R-O
AMSTE-TE-T (MR RITONDO)
ABERDEEN PROVING GROUND MD
21005-5006

CDR, US ARMY TROOP SUPPORT
COMMAND
ATTN: AMSTR-ME
AMSTR-S
AMSTR-E
AMSTR-WL
4300 GOODFELLOW BLVD
ST LOUIS MO 63120-1798

CDR
US ARMY NATICK RES & DEV CENTER
ATTN: STRNC-YE (DR KAPLAN)
STRNC-U
NATICK MA 01760-5000

CDR
US ARMY QUARTERMASTER SCHOOL
ATTN: ATSM-CD
ATSM-TD
ATSM-PFS (MR ELLIOTT)
FORT LEE VA 23801

CDR
US ARMY LOGISTICS CTR
ATTN: ATCL-MS (MR A MARSHALL)
ATCL-C
FORT LEE VA 23801-6000

PROJECT MANAGER
PETROLEUM & WATER LOGISTICS
ATTN: AMCPM-PWL
4300 GOODFELLOW BLVD
ST LOUIS MO 63120-1798

DIR
US ARMY MATERIALS TECHNOLOGY
LABORATORY
ATTN: SLCMT-M
SLCMT-EM (DR FOPIANO)
WATERTOWN MA 02172-2796

DEPARTMENT OF THE NAVY

CDR
NAVAL SEA SYSTEMS CMD
ATTN: CODE 05M4
WASHINGTON DC 20362-5101

CDR
DAVID TAYLOR NAVAL SHIP R&D CTR
ATTN: CODE 2830 (MR SINGERMAN)
CODE 2759 (MR STRUCKO)
CODE 2831
ANNAPOLIS MD 21402-5067

CDR
NAVAL RESEARCH LABORATORY
ATTN: CODE 6170
CODE 6180
CODE 6110 (DR HARVEY)
WASHINGTON DC 20375-5000

OFFICE OF THE CHIEF OF NAVAL
RESEARCH
ATTN: OCNR-126
CODE 432 (DR MILLER)
ARLINGTON, VA 22217-5000

CDR
NAVY PETROLEUM OFC
ATTN: CODE 43 (MR LONG)
CAMERON STATION
ALEXANDRIA VA 22304-6180

DEPARTMENT OF THE AIR FORCE

CDR
US AIR FORCE WRIGHT AERONAUTICAL
LAB
ATTN: AFWAL/POSF
AFWAL/POSL (MR JONES)
WRIGHT-PATTERSON AFB OH
45433-6563

HQ, USAF
ATTN: LEYSF (COL LEE)
WASHINGTON DC 20330

CDR
SAN ANTONIO AIR LOGISTICS
CTR
ATTN: SAALC/SFT (MR MAKRIS)
SAALC/MMPRR
KELLY AIR FORCE BASE TX 78241

HQ AIR FORCE SYSTEMS CMD
ATTN: AFSC/DLF
ANDREWS AFB MD 20334

1

1
1